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DEVELOPMENT OF IMPROVED ELECTROFORMING TECHNIQUE

Authored By:

Lee C. McCandless
Larry G. Davies

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Project Manager, John Kazaroff
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16. Abstract

The scope of this program was to experimentally develop techniques to reinforce or strengthen electroformed nickel to allow a fuller utilization of electroforming as a reliable and low cost fabrication technique for regenerately cooled trust chambers. Techniques for wire wrapping while electrodepositing were developed that can result in a structurally strong wall with less weight than a conventional electroformed wall. Also a technique of codepositing submicron sized ThO_2 particles with the nickel to form a dispersion strengthened structure was evaluated.

The standard nickel cylinders exhibited an average hoop strength of 80,000 psi with a yield strength of 65,000 psi and a modulus of 25.6×10^6 psi. The as produced dispersion strengthened nickel showed a hoop strength of 97,000 psi with a yield strength of 67,000 psi. This is an increase of 17,000 psi or 21% over the standard nickel hoop strength. The wire wrapped cylinders showed an increase strength over the standard nickel test samples of 26,000 to 66,800 psi which is in the range of 26 to 104% increase in strength over the base standard nickel. These latter test results are indicative of a volume percent wire reinforcement from 15 to 31. The measured hoop strength agree with calculated composite strengths based upon rule of mixtures.

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I - SUMMARY

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The standard nickel cylinders exhibited an average hoop strength of 80,000 psi with a yield strength of 65,000 psi and a modulus of 25.6 x 10^6 psi. The as produced dispersion strengthened nickel showed a hoop strength of 97,000 psi with a yield strength of 67,000 psi. This is an increase of 17,000 psi or 21% over the standard nickel hoop strength. The wire wrapped cylinders showed an increase in strength over the standard nickel test samples of 26,000 to 66,800 psi which is in the range of 26 to 104% increase in strength over the base standard nickel. These latter test results are indicative of a volume percent wire reinforcement from 15 to 31. The measured hoop strengths agree with calculated composite strengths based upon rule of mixtures.

II - INTRODUCTION

A. BACKGROUND

Various methods have been investigated in recent years to improve the fabrication of regeneratively cooled thrust chambers. One method that has been used successfully is electroforming. Thrust chambers have been built completely of electroformed nickel, including the inner wall, the coolant passage ribs, and the outer jacket. Other chambers have used electroforming for the outer jacket.

Although electroforming has been employed successfully, some problems have been encountered in its use. Some of these problems are non-uniform properties, non-reproducible properties, layer separation, nodule formation, and uneven build-up rates. These problems are, for the most part, solvable by better process control. Other shortcomings of the electrodeposition process are the relatively slow fabrication rates and the low strength of pure metals, such as nickel or copper, that may be easily electrodeposited.

To fully utilize electroforming as a reliable and low cost fabrication technique for thrust chambers, improvements over present capabilities must be obtained. Methods need to be developed to increase the strength of the deposited material. For example, techniques for wire wrapping while electrodepositing could be developed that would result in a strong wall of less weight than a conventional electroformed wall. Another reinforcing technique is the codeposition of submicron sized particles for dispersion strengthening. The major advantage of the wire reinforced electroform would be to provide yield strength properties comparable to the high nickel alloys without the corresponding fabrication difficulties of these alloys.

Similarly, one of the main advantages of electroformed dispersion strengthened nickel would be its improved elevated temperature strength, and resistance to recrystallization and creep at high temperatures. The use of reinforced material would not only produce lighter weight structures but might be expected also to reduce fabrication times compared to that of conventional electrodeposition.

B. OBJECTIVE

The objective of this program was to advance and improve the electroforming process by developing techniques to reinforce or strengthen the deposited material. The objective was accomplished by researching and evaluating methods to produce both dispersion-strengthened and wire-reinforced nickel cylindrical structures.

C. SCOPE OF WORK

The initial task was to verify the physical properties of an electroformed nickel from a specific bath formulation with target properties of 100,000 psi tensile strength and 10% elongation. Four nickel cylinders were then electroformed and burst tested in a hydraulic test assembly. The strength of the nickel electroform as determined by the pressure test and specimen tensile tests was used as a standard to compare against the results obtained with the strengthening techniques developed in Tasks II and III.

The objective of the second task was to improve the properties of the electro-deposited nickel through the use of co-deposited dispersion strengthening particles such as alumina and thoria. The preliminary work to perfect the process was done on a laboratory scale with small sample specimens. After the process was optimized with respect to tensile strength properties, four dispersion strengthened nickel cylinders were electroformed and burst tested to determine the improvement in strength properties that have been achieved as a result of the use of dispersion strengthening.

The third task was aimed at developing a method to fabricate a reinforced structure that is a combination of electroformed nickel and a high strength wire reinforcement. The wire reinforcement was a high strength stainless steel wire with an ultimate strength of 300,000 psi and a cross section designed to produce a uniform growth pattern which minimizes voids. When an optimum procedure was developed to produce the wire reinforced nickel electroform, nine cylinders were fabricated and burst tested to determine the changes in strength properties that have been achieved as a result of wire wrapping in conjunction with electro-deposition.

The burst strength or hoop tensile strength of the fabricated cylinders was measured by hydraulically pressurizing the inside wall of the cylinders while constraining the ends of the cylinder to insure a longitudinal failure in the cylinder wall. The hydraulic pressure at failure was recorded and the hoop tensile strength of the cylinder was calculated. The tensile properties of the electroformed nickel were determined by rupturing a machined tensile specimen in a Tinius Olsen testing machine.

III - EXPERIMENTAL PROCEDURE

A. STANDARD NICKEL ELECTROFORMING

The initial task was to achieve physical properties of an electroformed nickel material from a specific bath formulation with target properties of 100,000 psi tensile strength and 10% elongation. The verification of physical properties from a sulfamate - chloride bath was performed using flat plate tensile specimens machined from electroformed nickel sheet.

1. Standard Sulfamate Nickel Bath

A Barrett sulfamate nickel plating formulation was prepared in a fifty-five gallon tank for the initial phase of this contract. The average operating conditions for the process are as follows:

Temperature (°C)	50-55
pH	4.0
Density (°Be)	30
Current Density (asf)	50-60

The composition of the bath as received was analyzed to be the following:

Nickel Sulfamate	60 oz/gal
Nickel Metal	10.2 oz/gal
Boric Acid	3.0 oz/gal
Chloride	0.5 oz/gal
Anti-Pit Agent	0.05 oz/gal.

The initial formulation of the plating bath was analyzed and found to be low in boric acid content. Since the boric acid is difficult to dissolve, the required quantity was added over a period of two weeks, while the bath was dummy plated. Seven of the first ten plates from the bath were badly pitted due to the low boric acid. Most of these plates could not be tested because the tensile specimens were notched due to the pits. After the bath was worked for approximately 4800 amp-hours and the boric acid was adjusted to 5.75 oz/gal at 52°C, the physical properties of the bath were measured to be close to the target properties of 100,000 psi at 10% elongation. The electroforming tank is a 24 x 24 x 24 inch polypropylene plating tank equipped with stirrer agitation and a quartz immersion heater with thermostatic control. The anodes are rolled depolarized nickel bar housed in expanded mesh titanium anode baskets. The baskets are double bagged with dynel anode bags. The filtration is continuous through a Serfilco pump and filter unit. The plating power supply is a silicon control rectifier with ripple filter. An amp-hour meter is used to record the total cathode current used in the electroforming operation. Cathode current and voltage are displayed on calibrated meters.

Several processing variables that are critical to the success of producing uniform nickel deposits were monitored closely during the course of the study for the sulfamate-chloride baths used for cylinder fabrication.

a. Bath Sampling

The sample was taken by inserting a glass pipet about 0.5 inch in diameter into the solution with the top end uncovered. The solution will fill the tube as it is passed through the bath. When the tube reaches a certain depth the top is closed and the pipet withdrawn. The solution is transferred quickly to a sample container which is clean and dry and carefully identified as to tank number, date, and bath description. The sampling operation is repeated at different tank locations until sufficient sample has been obtained for all analyses and tests planned. Samples normally were taken when the solution level corresponded to the operating level of the bath.

b. Bath Analyses

Control of the plating bath during processing consisted of periodic chemical analysis to determine levels of an anion and cation concentration. Standard analytical procedures were utilized to ascertain whether or not the nickel, chloride, and boric acid concentrations were within specified limits. These control concentrations were maintained within the limits outlined earlier in this section. Analysis of each bath were conducted from samples taken by the standardized sampling technique described earlier. Samples were taken for analysis at the initial bath formulation and after roughly 5000 ampere hours of operation. Any time additions were made to the bath for corrective purposes a subsequent sample was taken for analysis to verify that all constituents have returned to within limits of the specifications.

c. pH

The hydrogen ion concentration is very important to the proper functioning of the bath. The control of the pH of plating solution is necessary to maintain the empirically determined acidity or alkalinity which has been shown to produce the best results. Appearance, stress, and leveling, for example may be affected adversely by operation of the bath outside the optimum pH range. High accuracy, however, is not usually necessary; measurements with a precision of ± 0.2 pH units are normally adequate.

The bath pH was measured with a Fisher Accumet pH Meter Model 210. This unit will provide an accurate, stable reading within 2 seconds and is reproducible to ± 0.02 pH. The allowable limits of variation for pH of the bath for meeting the specific objectives was determined to be for the sulfamate chloride bath a pH of 4.0 ± 0.2.

Measurement of pH was made on samples taken from the bath by the sampling technique described earlier. Measurements were made each morning and evening.

d. Stress of Nickel Deposits

Most electrodeposits of any metal are deposited in a state of stress usually tensile.

The need to avoid stress, which is relative with electroplating, is of great importance with electroforming. Precision electroforming needs plating at low stress, i.e., at conditions under which a spiral contractometer (passing current at the current density to be used) will not show a deflection to right or left in a period of at least twenty minutes. Quantitative calibration is not needed, but "low" means less than 1000 lb/in^2. The bath used in this program has tensile stresses on the order of 1000 to 10,000 psi. The bath can, however, be operated at zero or in the compressive stress state.

A spiral contractometer was used to measure stress. This device is based on the change of radius of curvature of a helix as the helix is plated. The stress in the deposit causes the helix to wind more tightly or to unwind, depending on whether the deposit stress is compressive or tensile. The change in radius of curvature is actually measured by the angular displacement of one end of the helix while the other end is held rigid. By means of gears the angular displacement is magnified and read on a dial as plating progresses.

e. Specific Gravity

A simple tank test of plating solutions was used for determination of the specific gravity. A hydrometer reading can indicate whether a solution is within concentration limits, whether stratification or incomplete mixing exist and whether decomposition products are building up. Because this test is easily and rapidly performed it was conducted on a twice daily basis during continuous bath usage. Significant variations in specific gravity were used as an indicator that more definitive bath analysis methods should be run immediately. Variation limits for specific gravity were determined to be as follows for a normal bath level:

Sulfamate Chloride Bath, 1.2 ± 0.1

f. Temperature

Temperature was daily monitored through all plating activitics. Use was made of a thermometer inserted directly into the bath. The allowable limits of temperature variation for the sulfamate chloride bath was 50 ± 2°C.

g. Bath Properties

Experimental work was conducted on producing flat plate test coupons from the production sulfamate-chloride bath for verification of the target properties of the electroform nickel. During the first 4800 amp-hours of operation with the bath, the properties of the nickel were a tensile strength of 100,000 psi or greater but an elongation of less than 10%. During the next 15,000 amp-hours of operation, the properties of the nickel electroformed from the bath changed to an elongation greater than 10% but a tensile strength less than 100,000 psi. In order to increase the tensile strength of the nickel, small additions of chloride ion were added to the bath to bring the level of chloride to 1.1 oz/gal.

Test samples of nickel from this bath condition showed that the target properties of 100,000 psi ultimate tensile strength and greater than 10% elongation were achieved. The data obtained for sample 243-21 tested by GTC and NASA-LEWIS is as follows:

GTC TEST RESULTS FOR SAMPLE 243-21

Test No.	Tensile Strength (10^3 psi)	Yield Strength (10^3 psi)	Elongation Gage-1 in. (%)	Elongation Gage-2 in. (%)
1	99.7	*–	*–	10.6
2	97.9	*–	*–	11.3
3	96.8	*–	*–	11.1
4	101	*–	*–	10.4
Average	98.9	–	–	10.9

*Not Tested

NASA-LEWIS TEST RESULTS FOR SAMPLE 243-21

Test No.	Tensile Strength (10^3 psi)	Yield Strength (10^3 psi)	Elongation Gage-1 in. (%)	Elongation Gage-2 in. (%)
1	101	69.5	17	-
2	100	69.6	16	-
3	101.2	66.9	16	-
Average	100.7	68.7	16.3	-
**4	51.2	6.45	47	-

**Annealed at 1500°f

Sample No. 243-23 was a preliminary electroformed nickel test cylinder which was used to test out the mechanical and electrical operations necessary to electroform the desired thickness of nickel on a removable cylindrical mandrel. The cylinder was electroformed to a total thickness of 0.090 inches, but was severely pitted on the lower one-third portion as well as over the entire surface of the test coupon mandrel.

Two nickel cylinders and supporting test samples were subsequently electroformed. The electroformed cylinders were not testable due to a large number of pits on the surface of the cylinder. The source of the pitting was found to be insufficient mechanical agitation and was corrected. However, before any additional test cylinders could be fabricated, a recirculating pump failure caused the loss of this entire bath. A new standard sulfamate-chloride bath was immediately formulated and put into operation. Additional flat plate test specimens were produced from this bath, which was designated P-SNCL-2, for verification of the target properties of the electroformed nickel. Test specimens number 243-34P and 243-39P showed properties which matched the target properties of the standard nickel. On the basis of the flat plate tensile specimens noted above, standard nickel cylinder fabrication was again begun.

2. Standard Nickel Cylinder Fabrication

A standard electroform nickel cylinder designated 243-42P was electroformed for 74 hours at a current density of 40 ASF in the P-SNCL-2 bath. The fabricated cylinder was of testable quality with no pitting on the surface. Unfortunately, the tensile test coupons which were attached to the cylinder showed the electroform nickel properties to be lower than the target properties. Additional cylinder fabrication was planned as soon as the cause of the low strength deposits on the test coupon mandrel was determined.

A set of double test plates, with the same surface area as the cylinder mandrel plus the test coupon mandrel, were submerged full depth in the bath. Based on the results of these test plates, a larger capacity filtering system capable of pumping 760 gallons per hour was installed on the 55 gallon production bath. Also, the anode baskets were lengthened from 18 inches to 24 inches to provide full depth anode exposure. As a result of these changes, the variability in mechanical properties of the electroformed nickel throughout the bath was reduced to within experimental error of the test measurements.

During the fifth and sixth months of the contract, the four standard nickel electroform cylinders were produced. The experimental fabrication conditions are given in Table I. All four of these cylinders were to be burst tested, two in the as-produced condition and two in a heat treated condition. The heat treatment consisted of heating in an inert atmosphere to a temperature of 1500°F (815°C), then holding at temperature for 1 hour and air cooling.

TABLE I.

Standard Nickel Cylinders Produced from Sulfamate-Chloride Bath

Cylinder No.	Sample No.	Current Density (asf)	pH	Bath Temp. (°C)	Plating Time (Hrs.)	Plated Wall Thickness (Mils)	Hoop Tensile Strength Ksi
7	243-88P2M	20	4.2	49	91	71	84.1
8	243-93P2M	20	4.0	49	87	90	75.7
10 Heat Treated*	243-100P2M	20	4.1	49	75	71	63.5
12 Heat Treated*	243-139P2M	20	4.1	49	62	56	44.8

*Heat treated at 1500°F (815°C) for 1 hour and slow cooled.

The strength of the electroform cylinders as determined by the hydraulic pressure test and specimen tensile tests will be used as a standard to compare against the results obtained with the strengthening techniques developed in Tasks II and III summarized in Section E.

3. Electroform Mandrel Design

Withdrawal of a precision mandrel surface from within an electroformed nickel cylinder of 4-inch ID and 8-inch length without damage to the mandrel or cylinder requires a mandrel design of some collapsible type. A standard collapsible mandrel of metal, made in sections, is not desirable since it does not provide a smooth, uninterrupted surface for electroforming. The sort of design that has been found successful and convenient for electroforming cylindrical sections is a stainless steel mandrel core covered by a layer of low temperature melting alloy, upon whose surface is electro-formed the cylinder. Collapsing of the mandrel is accomplished by heating to the melting point, 281°F, and allowing the molten alloy to run out. The steel core can then be simply withdrawn and the cylinder is left freestanding.

4. Mandrel Casting

The completed collapsible electroform mandrel together with the casting mold and heaters is shown in Figure 1. The mandrel is formed by positioning a 3-1/2 inch diameter knurled stainless steel core into a 4-1/8 inch diameter split casting mold and cast with the low melting alloy. The alloy is first heated to a temperature of approximately 300°F, then poured in a molten state into the heated casting mold. The band heaters on the casting mold are turned off and the entire assembly is allowed to cool slowly to avoid entrapped air pockets. After cooling, the cast mandrel is removed from the split mold and then machined to a diameter of 4.000 ± .005

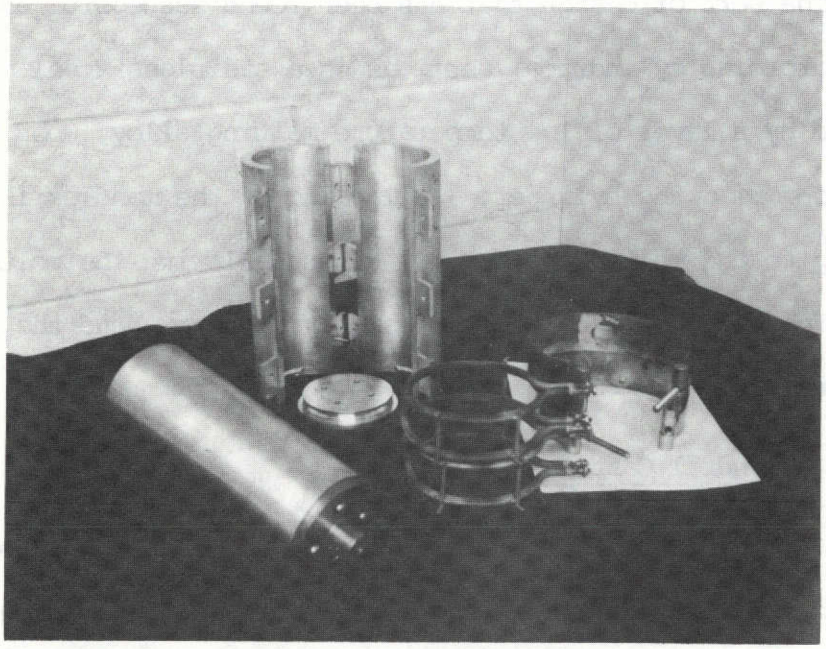


Figure 1. Collapsible Metal Electroform Mandrel With Casting Mold and Heaters

inches with a 32 rms finish. The mandrel is then ready for electroforming. Two polypropylene shields are attached to the top and bottom of the mandrel to prevent excessive build-up in these areas. A one inch diameter stainless steel shaft is attached to the top of the mandrel for rotation purposes. Attached to the bottom of the electroform mandrel is a test coupon mandrel comprising a stainless steel block 2-1/2 inches on a side by 6-1/2 inches long. The entire mandrel assembly is shown in Figure 2.

The cast cylindrical mandrel is prepared for plating by degreasing, dipping in 10% fluoboric acid solution and water rinsing in tap then deionized water and plating. The test coupon mandrel is prepared by degreasing, acid dipping, water rinsing and plating.

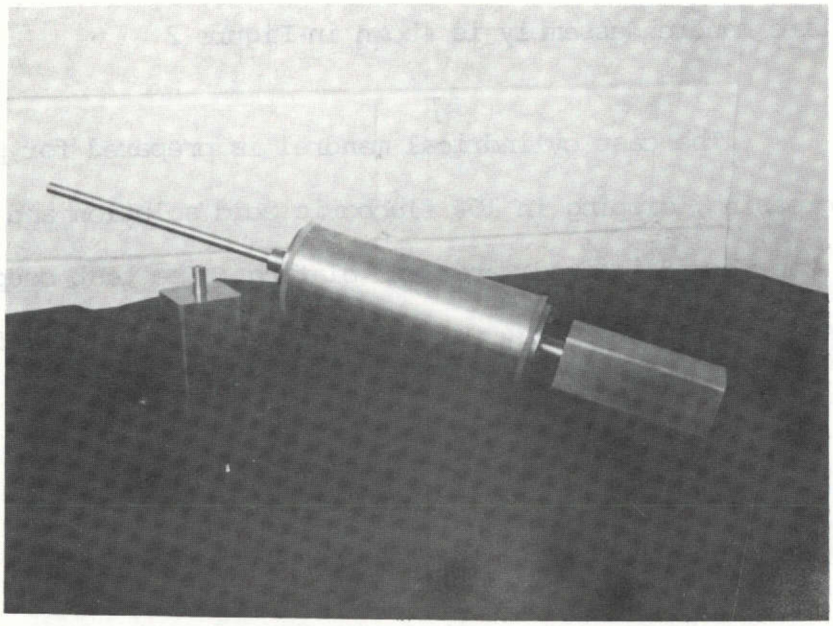


Figure 2. Metal Electroform Cylinder Mandrel With Verification Sample Mandrel Attached

B. DISPERSION STRENGTHENING STUDIES

The objective of the second task was to improve the properties of the electro-deposited nickel through the use of co-deposited dispersion strengthening particles such as alumina and thoria. The preliminary work to perfect the process was done on a laboratory scale with small sample specimens. After the process was optimized with respect to tensile strength properties, four dispersion strengthed nickel cylinders were electroformed and two were burst tested to determine the improvement in strength properties that had been achieved.

1. Experimental Investigation

Two experimental five gallon nickel sulfate-chloride baths were used for the initial portion of the dispersion strengthening studies. Nickel plated from both baths were made into flat plate tensile specimens for verification of the target standard nickel properties before being operated with dispersion particles.

Aluminum oxide particles were dispersed in one of the baths. The Al_2O_3 particles used in this study were polishing grade gamma Al_2O_3 with an average particle size of 0.05 micron. A concentration of Al_2O_3 equal to 16 g/l in the 5 gallon bath was added to two liters of the bath and ball milled for 48 hours to achieve complete dispersion in the bath. The pH of the dispersion was tested and adjusted if necessary before adding to the plating bath. An air agitation system was used to maintain the dispersion in the cathode compartment of the bath. Subsequently, a concentration of Al_2O_3 equal to 25 g/l in the bath was prepared and added to the bath.

Thorium oxide particles were dispersed in the other experimental bath. The ThO_2 particles being used in this study have a particle range of 100 to 300 angstroms. A concentration of ThO_2 equal to 5 g/l in the 5 gallon bath was added to one liter of distilled water and ball milled for 48 hours to achieve a completely dispersed hydrated material, which was then added to the sulfamate-chloride bath. An air agitation system was used to maintain the dispersion.

Both experimental particle baths were operated with a membrane separating the anode compartment from the cathode compartment. The compartment was separated by a porous paper membrane that keeps the particles out of the anode compartment and prevents anode sludge from getting into the cathode area to affect the deposit. However, a number of difficulties were experienced with rupture of the membrane and leakage of the particles into the anode compartment. Also the necessary plating current and voltages had to be greatly increased because of a voltage drop across the membrane. All of these factors combined led to discontinuing the use of the membrane during the cylinder fabrication.

2. Dispersion Bath Experimental Parameters

The experimental parameters which were investigated for the formation of dispersion strengthened electroformed nickel included:

a. Bath particle concentration;

b. Chemical composition of particle dispersion;

c. Dispersion particle size;

d. Cathode current density.

The plating conditions of the dispersion experimental baths were maintained as close as possible to the normal operating conditions of the standard sulfamate nickel bath. However, the membrane separation of anode and cathode compartments provided difficulty with control of the pH, temperature and particle concentration parameters. The experimental bath plating parameters for the dispersion studies is shown in Table II.

Samples from each bath were machined into standard tensile specimens and tested for tensile strength and elongation at room temperature and 1500°f.

TABLE II

Experimental Plating Parameters for Dispersion Strengthening Studies

Aluminum Oxide Dispersion

Sample No.	Current Density asf	pH Anode	pH Cathode	Temperature (°C) Anode	Temperature (°C) Cathode	Particle Concentration in Bath (g/l)	Cathode Amp-Hours	Thickness Mils
76E2	20	3.9	4.0		50	16	350	37
86E2	20	5.5	4.5		49	16	1530	92
92E2	40	3.8	4.0	44	49	16	360	
97E2	20	4.4	4.5	38	49	10	1600	98
108E2	20	4.8	4.0	36	49	16	320	43
113E2	20	4.5	4.1	36	50	25	480	47
120E2	40	4.9	4.0	36	49	25	720	61
126E2	60	4.7	4.1	35	50	25	705	69

Thorium Oxide Dispersion

Sample No.	Current Density asf	pH Anode	pH Cathode	Temperature (°C) Anode	Temperature (°C) Cathode	Particle Concentration in Bath (g/l)	Cathode Amp-Hours	Thickness Mils
92E3	20	3.1	4.0	44	49	5	360	35
100E3	20	4.3	4.3	39	49	5	490	34
106E3	30	4.8	4.0	44	51	5	480	41
108E3	40	4.3	4.3	44	49	3.7	640	54
127E3	50	4.2	4.0	39	46	5	569	59

3. Experimental Dispersion Strengthened Test Results

The room temperature and 1500°F tensile strength and elongation date for Al_2O_3 test samples is given below:

Sample No.	Current Density (asf)	Particle Concentration in Bath (g/l)	Avg. Tensile Strength 10^3psi		Avg. Elongation in 2 in. Gauge %	
			Rm. Temp.	1500°F	Rm. Temp.	1500°F
76E2	20	16	129	6.1	6.5	12.3
86E2	20	16	97.5	11.0	6.4	5.8
92E2	40	16	81.4	7.7	10.1	4.2
97E2	20	10	111	8.6	4.5	2.4
108E2	20	16	114	6.1	9.7	10.4
113E2	20	25	117	4.2	1.1	10.4
120E2	40	25	105	5.7	2.0	6.8
126E2	60	25	84.7	7.9	6.3	5.4
128E2	30	25	95.0	5.9	2.9	5.3

The room temperature and 1500°F tensile strength and elongation data for ThO_2 test samples is given below:

Sample No.	Current Density (asf)	Particle Concentration in Bath (g/l)	Avg. Tensile Strength 10 psi		Avg. Elongation in 2 in. Gauge %	
			Rm. Temp.	1500°F	Rm. Temp.	1500°F
92E3	20	5	138	8.0	7.3	9.7
100E3	20	5	124	7.8	3.9	11.3
106E3	30	5	130	5.9	4.1	8.6
108E3	40	3.7	127	6.6	3.4	6.7
127E3	50	5	107	5.1	5.2	8.4
129E3	20	5	129	4.1	3.4	14.1

Figure 3 shows a dispersion of ThO_2 particles in one of the electroformed nickel test coupons. The figure was made by electron microscopy replica techniques. Figure 4 shows a dispersion of Al_2O_3 particles in one of the electroformed nickel test coupons by an electron replica photomicrograph.

The room temperature tensile strength properties of the alumina dispersed nickel show an average strength approximately equal to the standard nickel at the lower current density (20 asf) with a subsequent decrease in strength at higher current densities. This trend is seen with two particle concentrations in the bath; i.e., 16 and 25 grams per liter. The 1500°F tensile data shows a large deviation in strength values from 40 percent below that of the standard nickel to 15 percent above. This data is from a limited number of samples, but the data indicates less reproducibility with this material than with the thoria dispersion. The room temperature tensile strength properties of the thoria dispersed nickel show a 7 to 10 percent increase in strength over the standard nickel at a 20 to 30 asf current density, with no increase in strength at a higher current density (50 asf). However, tensile strength measurements at 1500°F (4 samples with two tests each) show a decrease of 0 to 20 percent below the standard nickel. Although this data is from a limited number of samples, a conclusion was drawn at the time with regard to the thoria system. The room temperature tensile strengths are higher than those for the standard nickel or for the alumina dispersed nickel, the high temperature data is also more consistant than the alumina dispersion, although the alumina data has two samples which had a higher strength. On

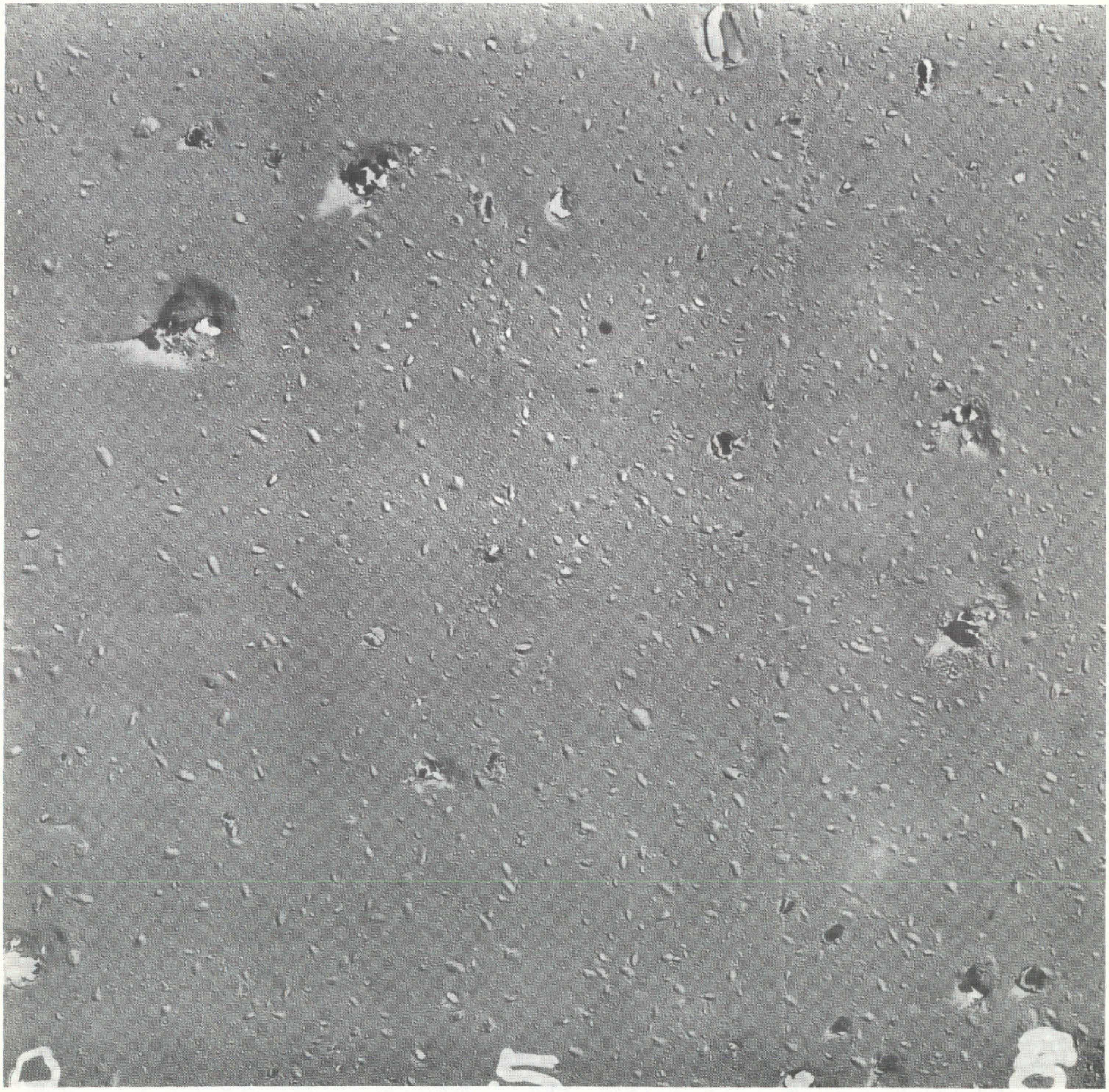


Figure 3. Electron Replica Photomicrograph of ThO_2 Particles Dispersed in ED Nickel, 7700X

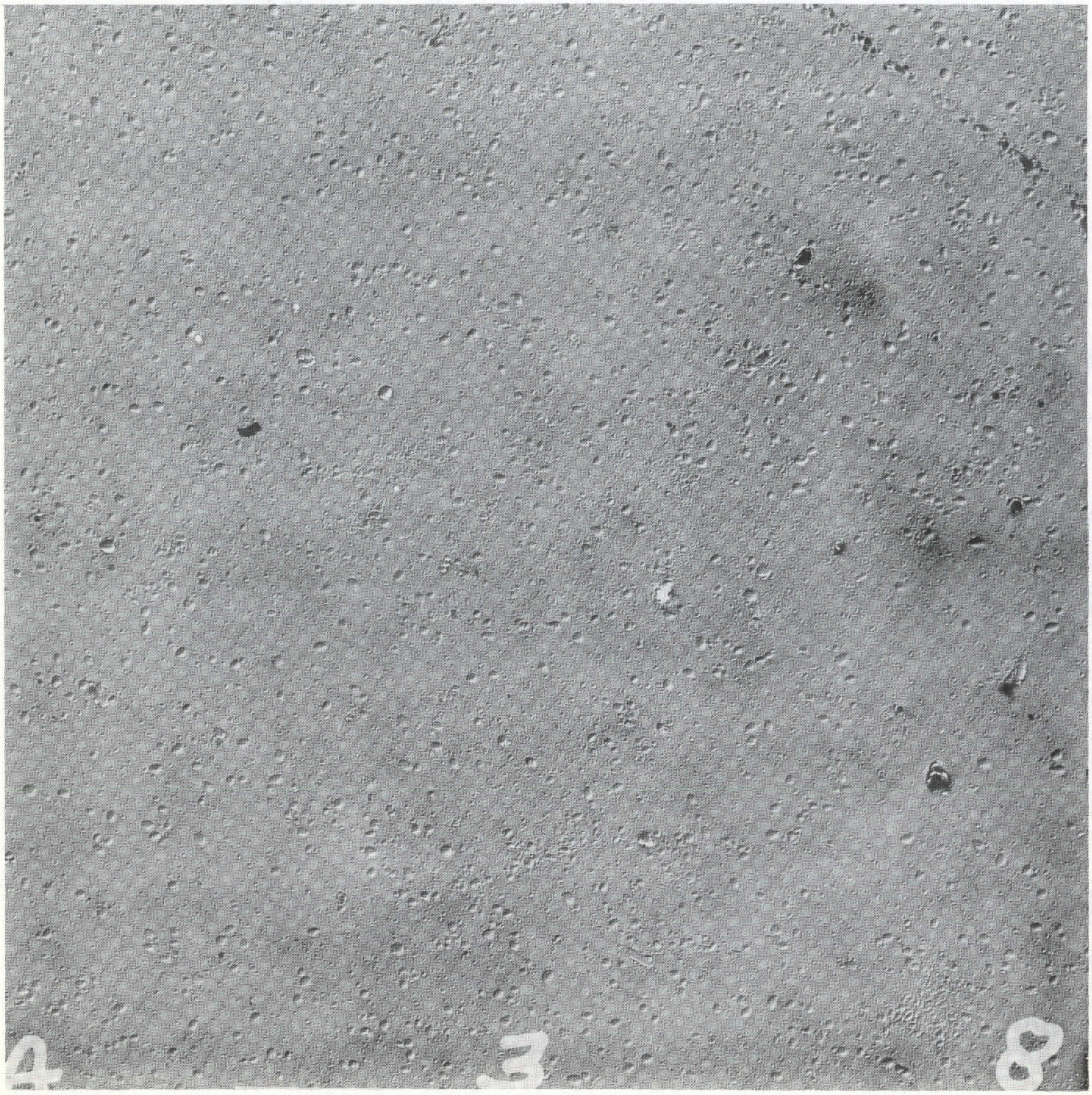


Figure 4. Electron Replica Photomicrograph of Al_2O_3 Particles Dispersed in ED Nickel, 7700X

the whole, it was concluded that the thoria dispersion at its current stage of development was the better of the two dispersion strengthened materials. Thus the thoria dispersion was used for fabrication of cylinders.

4. Dispersion Strengthended Cylinder Fabrication

a. Procedure

A separate fifty-five gallon electroforming tank was placed in operation for this phase of the program. The plating tank was a 24 x 24 x 30 inch polypropylene tank equipped with compressed air agitation, quartz immersion heaters and a thermostatic heater control. The anodes were rolled depolarized nickel bar housed in expanded mesh titanium anode baskets. The baskets were double bagged with dynel anode baths. Filtration of the bath was done on an intermittent basis after the agitation was stopped for a period of time. A phosphate type anti-pit agent compatible with the air agitation was used in a sulfamate-chloride bath formulated by adding chloride ion to a standard Barrett-Sulfamate nickel plating formulation.

The bath was dummy plated and the desired quantity of boric acid was added to the bath over a two-week period. After the physical properties of the bath were measured to be close to the target properties of 100,000 psi at 10% elongation, a quantity of the 100-300 Å thorium oxide was added to the bath equivalent to 5 g/l concentration. The cylinder fabrication was accomplished in an identical manner to that of the standard nickel cylinders described previously.

b. Cylinder Fabrication Parameters

Four dispersion strengthened nickel cylinders were electroformed from the thorium oxide dispersion bath. The fabrication parameters are listed in Table III.

TABLE III

Dispersion Strengthened Nickel Cylinders Produced from Thoria Sulfamate-Chloride Bath

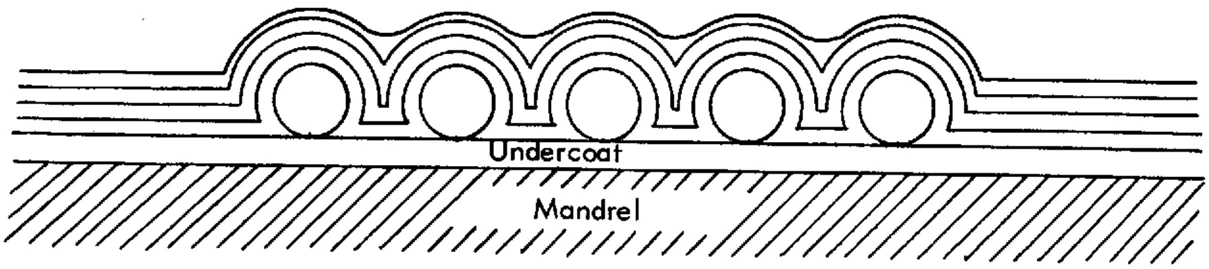
Cylinder No.	Sample No.	Current Density (asf)	pH	Bath Temp. (°C)	Plating Time (Hrs.)	Plated Wall Thickness (Mils)	Hoop Tensile Strength psi
17	246-2P3M	20	3.9	48	79	70	102,000
18	246-3P3M	20	4.1	48	81	71	93,000
20	246-6P3M	20	4.1	48	72	68	Not tested
21	246-6P3M	20	4.1	48	67	70	Not tested

C. WIRE WRAPPED STRENGTHENING STUDIES

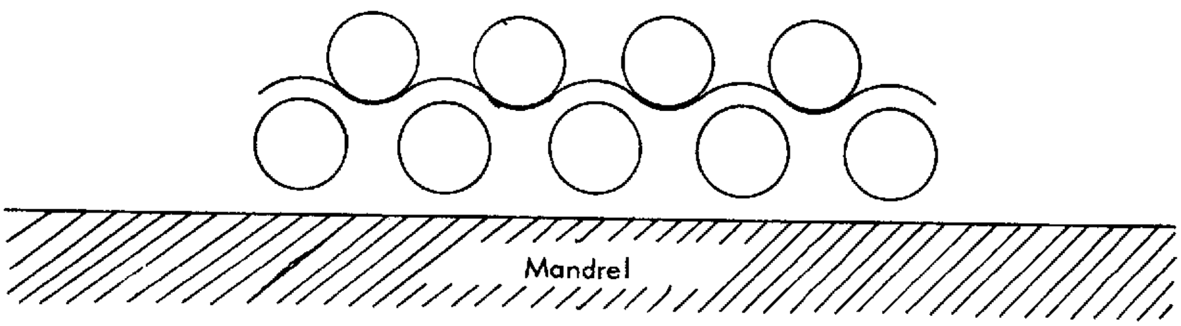
1. Background

Continuous steel wire hoop-wound into an electroformed nickel matrix thin-walled cylinder is an example of an undirectional array ductile-ductile composite material. The expected strength of a fibrous composite material is approximately equal to the rule of mixture value based upon the volume and strength of the constituent materials.

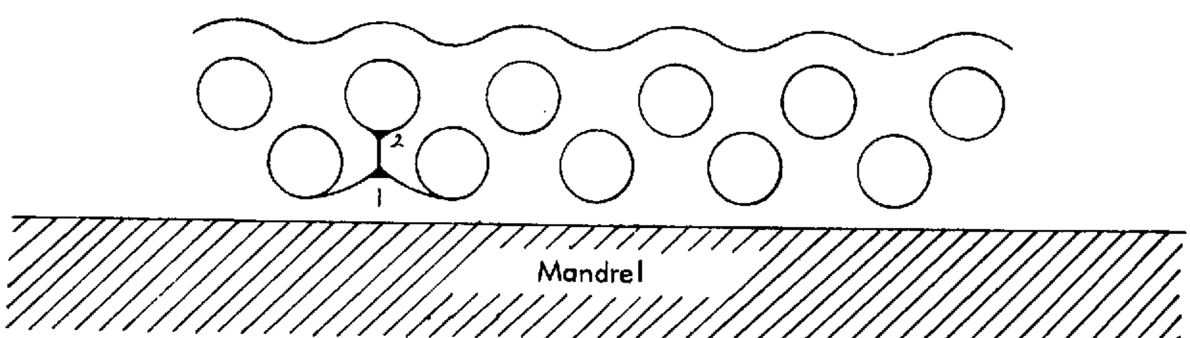
Early investigations into electroforming continuous reinforcement fibrous composites identified a problem of a void area occurring with the electroform growth pattern around a round wire reinforcement and the surface of the mandrel or previous layer. The void is found at the intersecting boundary between the matrix growth from the flat surface and the outer surface of the reinforcement as shown in the schematic representation illustrated in Figure 5. This problem is more severe with round reinforcements than with other shaped reinforcements. Since the throwing power of most electroforming baths is less than ideal, the recessed regions, i.e., the point of fiber matrix intersection will have less metal deposited than on the remainder of the surface. The degree of metal deposition in the recessed region is ultimately related to the thickness of the cathode diffusion layer. If irregularities occur on the cathode surface, the diffusion layer will vary in thickness with the result that the rate of mass transport of the depositing ions will be less in the recessed region. Thus, the deposit will not



A. Monolayer Growth



B. Multilayer Formation



C. Potential Void Sites

Figure 5. Schematic Representation of Electroformed Composite Formation.

reproduce the contours of the cathode surface satisfactorily.

In order to eliminate this problem a special reinforcement cross section was developed that would readily permit fill by the electroformed nickel without voids due to the growth patterns between the mandrel surface and the reinforcement.

2. Reinforcing Wire Selection

Two sizes of type 302 stainless steel wire were ordered for the wire reinforcing phase of the program. This material can best be described as half-round wire with nominal dimensions of 0.004 by 0.008 inches and 0.010 by 0.020 inches. A series of measurements made on the actual wire showed that, for the small size wire, the width was an average of 8 mils, while the height was 4.5 mils; while for the large size wire, the width was an average of 19.3 mils, while the height was 10.4 mils.

The tensile strength of the wire was measured over a six inch gauge length. The tensile strength of the large wire was 277,000 psi while the small wire exhibited a tensile strength of 390,000 psi.

3. Experimental Wire Reinforcing Studies

The standard nickel sulfamate-chloride bath used for Phase I of the study was also used for the wire reinforcing phase. During the experimental portion of this phase, the wire was wrapped on small diameter cylindrical mandrels to determine the spacing and bonding characteristics necessary to optimize the process. The spacing which was used included nominal spacing, one diameter and two diameters apart. Small diameter wound cylinders were plated with nickel to determine the fill between the wires. Cross sections of the wound cylinders were made and are presented in Figures 6 through 9. In all cases with the exception of the

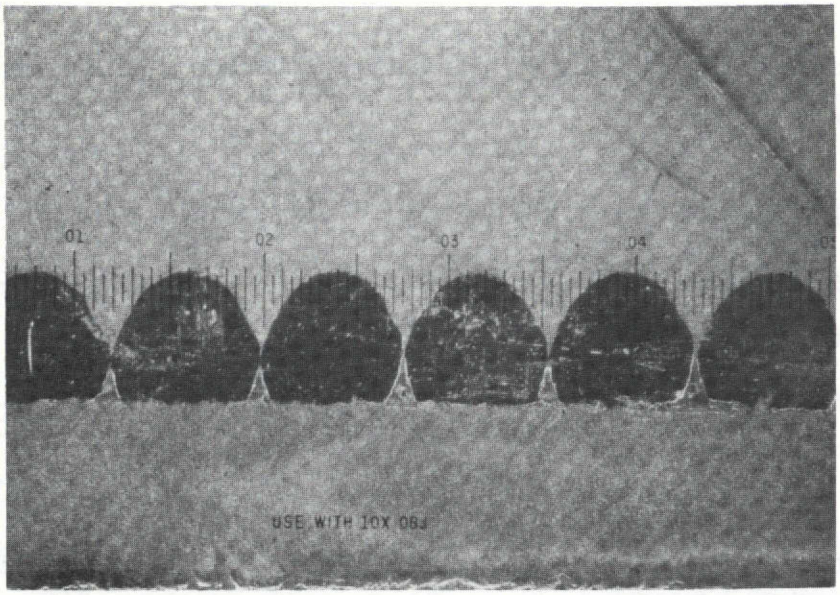


Figure 6. Reinforcement of Stainless Steel Wire in Electroformed Nickel Showing Fill with 8 mil Shaped Wire at Nominal Spacing, 100X.

297-15-2

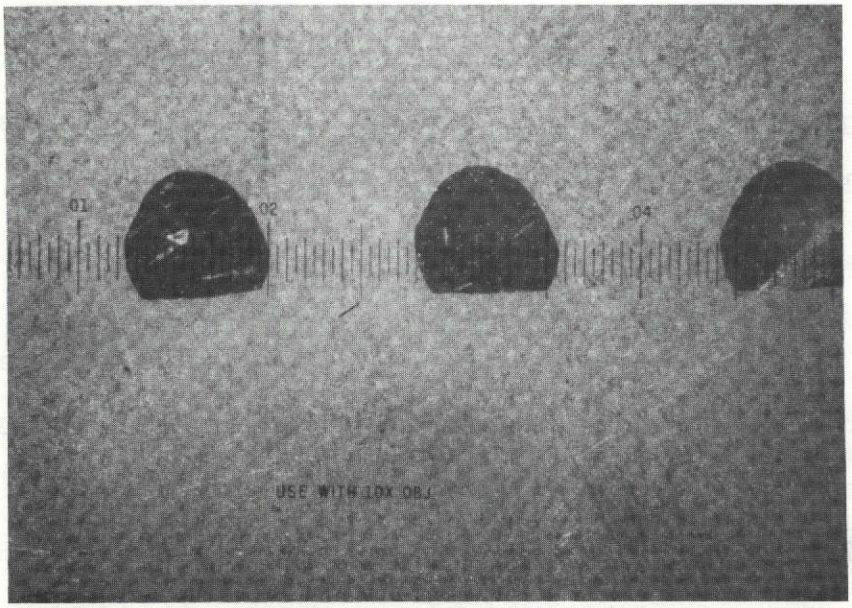


Figure 7. Reinforcement of Stainless Steel Wire in Electroformed Nickel Showing Fill with 8 mil Shaped Wire at One Diameter Spacing, 100X.

297-15-3

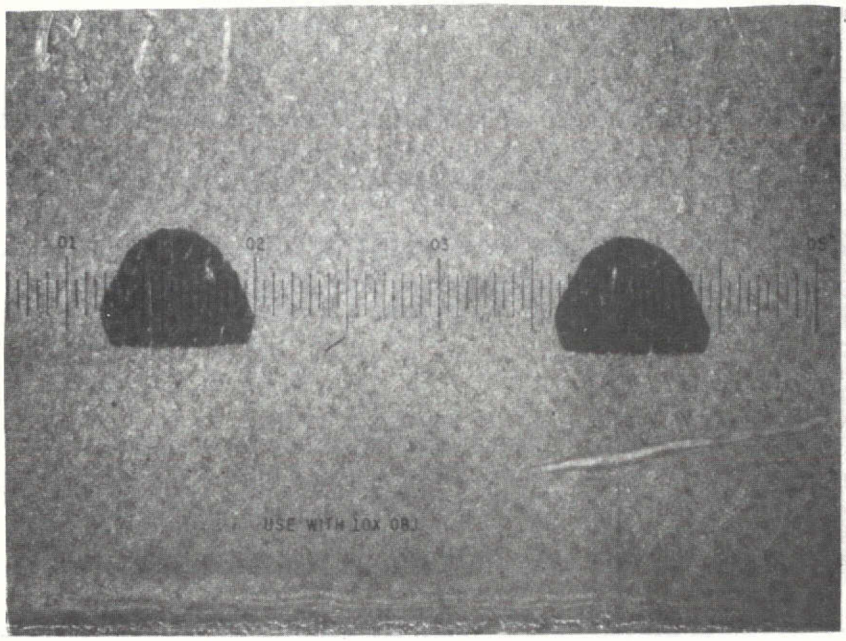


Figure 8. Reinforcement of Stainless Steel Wire in Electroformed Nickel Showing Fill with 8 mil Shaped Wire at Two Diameters Spacing, 100X.

297-15-4

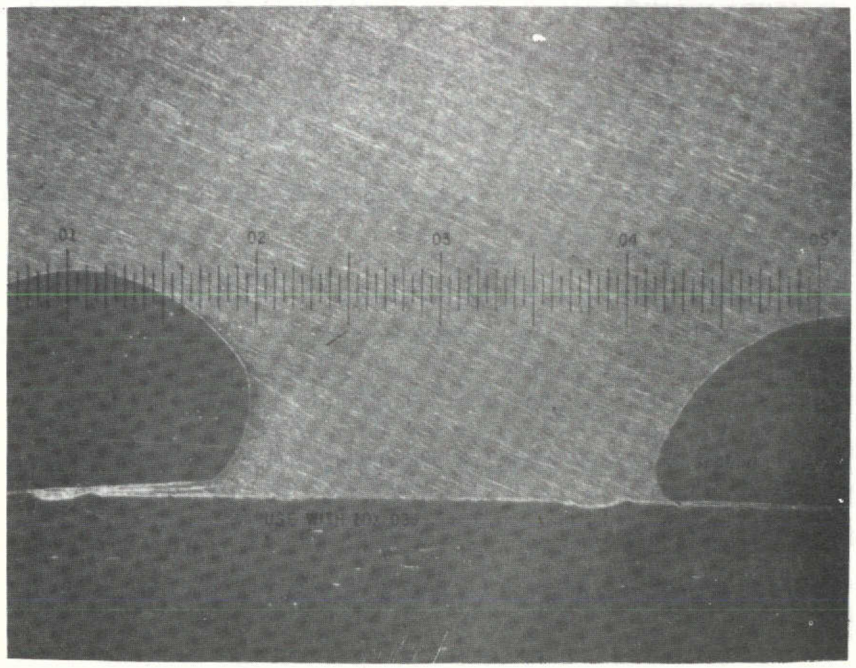


Figure 9. Reinforcement of Stainless Steel Wire in Electroformed Nickel Showing Fill with 20 mil Shaped Wire at One Diameter Spacing, 100X.

nominal spacing, the nickel fill is uniform, in intimate contact with the wire and without any void areas between the wires. These results indicated that the fabrication of wire wound cylinders should be accomplished with half diameter and one diameter spacing.

4. Procedure for Wire-Wrapping Cylinder Fabrication

The general procedure that was used for the Task III wire winding and compositing effort was influenced by the filling and leveling behavior that was found in previous work on electroforming nickel over square wire arrays. A completely smooth and cylindrical overplate of several mils thickness on top of an array of square wires is essentially impossible to obtain. If this surface were not flattened, the next layer of wire winding and overplating would be progressively more non-uniform, disrupting the wire spacing and creating additional matrix void areas. For this reason the nickel overplate in each layer is well beyond that required and is then machined back to a smooth surface of the required thickness. The winding procedure was:

a. electroform the base layer upon the mandrel surface;
b. attach and wind the wire at the required spacing under sufficient tension to keep good contact with underlayer;
c. overplate with nickel to fill gaps and plate a few mils of nickel in excess at the bottoms of the corrugations in the overplate surface to allow for machining to a smooth surface;

d. remove from bath, rinse and machine back to required surface and thickness;

e. wind next layer of wire;

f. go through surface degreasing and reactivation procedure;

g. overplate as in 3 above and continue in like fashion until last required layer is obtained.

The wire winding was carried out with an unspooling and gear drive spacing apparatus provided by a precision lathe.

5. Activation Procedure for Plating Nickel on Wire Wound Nickel Cylinders

In order to replate nickel onto a plated nickel surface, the surface must be activated to prevent delamination of the subsequent plate. Any interruption in current during plating or removal of the part from the bath will cause the nickel surface to become passive. This is particularly true when the part was removed for machining or grinding operations which were necessary in this program. After machining and winding, the nickel activation and plating procedure was as follows:

a. Degrease

b. Soak clean in commercial alkali-cyanide cleaner for 1-1/2 minutes

c. Water rinse

d. Activate in nickel potassium cyanide solution for 30 seconds

e. Water rinse

f. Nickel strike in woods nickel bath for 1-1/2 minutes

g. Water rinse

It is also worthy to note the above described procedure was necessary for preparing stainless steel for adherent plating. Therefore when the nickel is activated, the steel wire will also be activated such that good bonding will be obtained on the steel wires as well as the previous nickel layer.

The reinforcing wire was attached to either end of the mandrel to tie down the ends for overplating. One wire end was tied down at the start of the winding of a given layer and the other end at the completion, by means of plastic screws to minimize dendrite formation at the edges during overplating.

The schedules that were followed in winding the various cylindrical laminate forms are given in Tables IV, V, and VI. These specify the wire spacings and thickness of the alternating layers. The fabrication parameters for the wire-wrapped nickel cylinders produced from the sulfamate-chloride bath is given in Table VII.

Table IV. Wire Spacing Schedules

Number of Cylinders	Wire Width (inches)	Space Between Wires (inches)
2	0.020	0.020
3	0.020	0.010
3	0.008	0.008
1	0.020	0.010 (Four Wire Layers)

Table V. Nominal Lamination Schedule, 0.010 inch Height Wire

Layer	Thickness (inches)	Material
1	0.015	electrodeposited nickel
2	0.010	wire
3	0.015	nickel over the wire*
4	0.010	wire
5	0.020	nickel over the wire*

Table VI. Nominal Lamination Schedule, 0.004 inch Height Wire

Layer	Thickness (inches)	Material
1	0.010	electrodeposited nickel
2	0.004	wire
3	0.008	nickel over the wire*
4	0.004	wire
5	0.008	nickel over the wire*
6	0.004	wire
7	0.008	nickel over the wire*
8	0.004	wire
9	0.020	nickel over the wire*

*nickel to fill space between wires.

TABLE VII

Wire Wrapped Nickel Cylinders Produced from Sulfamate-Chloride Bath

Wire Size (Mils)	No. of Wire Layers	Cylinder No.	Sample No.	Current Density (asf)	pH	Bath Temp. (°C)	Plating Time (Hrs.)	Plated Wall Thickness (Mils)	Hoop Tensile Strength psi
20	2	9	243-99PM	20	4.1	49	51	81	126,000
20	2	13	243-143PM	20	4.2	49	56	62	125,000
20	2	14	243-144PM	20	4.2	49	97	62.5	99,200
20	2	15	243-146PM	20	4.2	49	123	61	131,000
8	4	16	243-148PM	20	4.1	49	177	70	Not tested
8	4	19	246-4PM	20	4.1	49	254	70	147,600
20	4	22	246-7PM	20	4.1	49	—	—	Not tested
20	2	23	246-8PM	20	4.1	48	—	—	Not tested
8	3	24	246-9PM	20	4.1	49	—	—	Not tested

D. PHYSICAL PROPERTY TESTING

The physical property testing of the nickel electroformed material was a major integral function of the program. The tensile properties of the electroformed nickel were determined by rupturing a machined tensile specimen in a Tinus Olsen testing machine, while an extensometer was used to generate a stress-strain curve. The burst strength or hoop tensile strength of the fabricated cylinders was measured by hydraulically pressurizing the inside of the cylinders to rupture of the cylinder wall. Strain gages were attached to the outer wall of the cylinder and a stress strain plot to the limits of the gage was obtained. The hydraulic pressure at failure was recorded and the hoop tensile strength of the cylinder was calculated.

1. Tensile Strength, Yield Strength, Elongation and Modulus at Room Temperature and 1500°F.

The data is generated at room temperature from a stress-strain plot of the specific nickel sample being tested. The tensile test coupons, six inches in length, one-half inch in width, reduced to one-quarter inch over a two inch gage section, were tested on a Tinus Olsen 12,000 pound Electromatic test machine. A clamp-on microformer type extensometer was used to generate the stress-strain curve. One type of extensometer mounting is shown in Figure 10, while the test machine is shown in Figure 11. The particular extensometer shown in the above figure, was designed primarily for high temperature measurements, and has only a one inch gage length. The contract called for measurement of elongation over a 2-inch gage length. Unfortunately, room temperature elongation values obtained with the one inch high temperature-type-extensometer do not compare directly with a method using two inch gage marks. This difference is due to the occurrence of

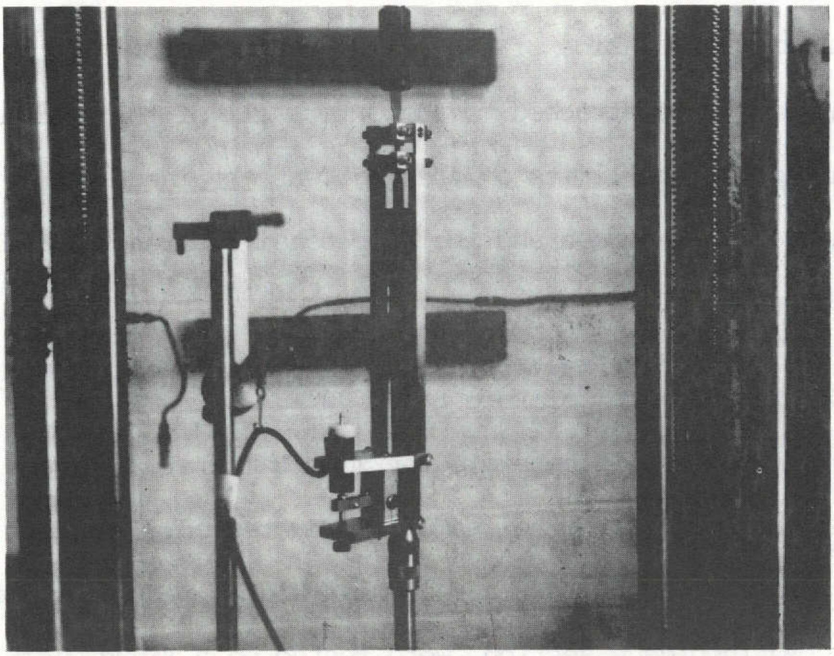


Figure 10. Tensile Testing Set-up Using High Temperature Type Extensometer

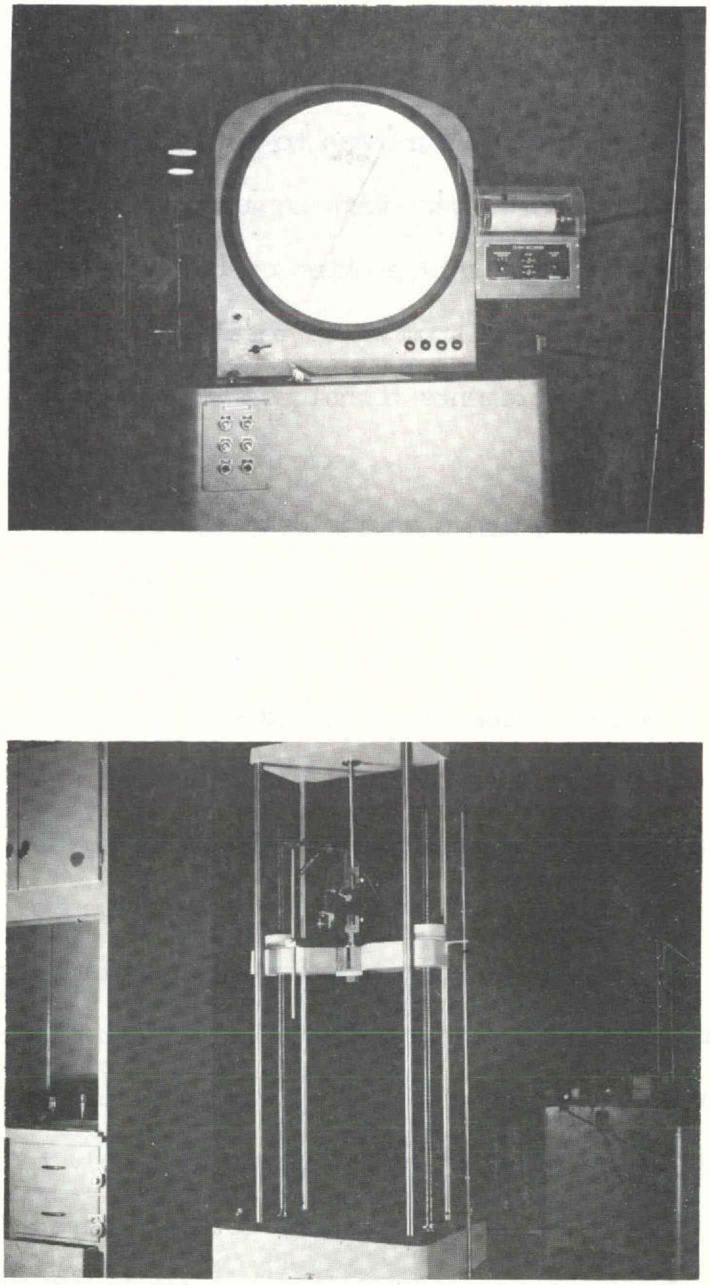


Figure 11. Universal Testing Machine

localized reduction in area or "necking" which occurs just before failure and which is more pronounced in the one inch gage measurement. When "necking" occurs, elongation values are not independent of gage length.

The initial program procedure was to measure total elongation using two methods; measurement of gage mark separation using a 2 inch gage, and the use of a microformer-type extensometer having a one inch gage to generate a stress-strain curve from which total elongation would be obtained. Both of these measurements techniques were to have been used at room temperature and 1500°F (815°C), the extensometer method being preferred because it includes in the total elongation data the elastic or Hook's Law elongation while the gage mark method does not. For the specimens measured to date this amounts to a total elongation value (by the gage mark method) about 0.3% low. Some difficulties were later encountered with the extensometer measurements of total elongation because of the extreme reduction in area of the specimen after high elongation. As a result of the area reduction, the extensometer clamps became loose and the extensometer slipped. At room temperature the clamps could be tightened by hand as the test progresses but this was not practical at 1500°F. For this reason the gage mark method was used for total elongation at 1500°F. The high temperature extensometer was used only to collect modulus and yield strength data which could be obtained before high elongation takes place. While the gage mark method may not be the optimum measurement technique, it is an acceptable method as per ASTM Tension Testing of Metallic Materials (E8). The method was particularly suitable for this program where comparable results are required at room temperature and 1500°F.

During the latter portion of the program, a two-inch gage extensometer was obtained and room temperature stress-strain curves were produced. The elongation values obtained at room temperature with the extensometer and with the gage marks agreed well within the experimental error from one measurement to another.

2. Burst Test Apparatus

The cylinders fabricated in each of the three tasks were pressure tested to failure using the second of the burst test apparatus described below.

a. Non Constrained End Test Apparatus

This apparatus consisted of an internally pressurized hydraulic cell, with the cylinder to be tested mounted inside between a top and bottom closure. The closures were designed with gasket and "O" ring seals designed to seal to the inside surface of the cylinder. The hydraulic pressure was applied on a moveable piston mounted through the top closure by the Tinius Olsen Electromatic Testing Machine. This apparatus worked well when checked out using a steel cylinder machined from a solid bar of type 1116 free machining carbon steel. However, if the test cylinder is sufficiently ductile like electroformed nickel, the cylinder wall deforms and the hydraulic fluid can leak past the end closure seals. The test worked well for the carbon steel cylinder because it exhibited brittle failure, but was not successful in bursting to failure a nickel electroform cylinder.

b. Constrained End Test Apparatus

The problems associated with burst testing a ductile metal with a high tensile strength such as electroformed nickel necessitated using a second completely different test apparatus from that described previously. This apparatus shown in Figure 12 used mechanical end seal caps which

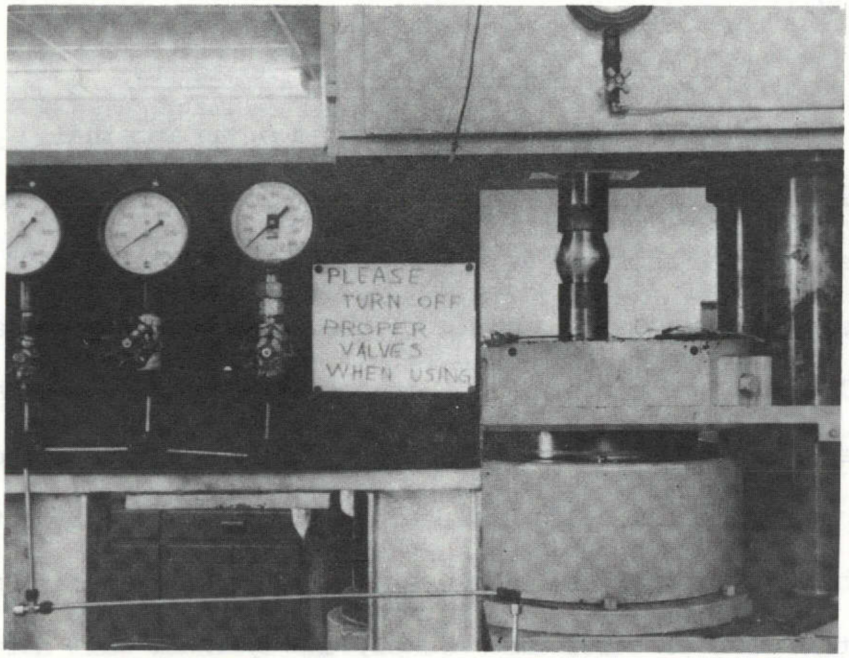


Figure 12. Constrained End Hydraulic Burst Test Apparatus

were epoxy bonded to the cylinder ends. The end seals are held in a constrained fixed position in between two platens of a 20 ton hydraulic press to prevent failure of the end seals due to internal pressure.

The cylinders were pressurized by pumping hydraulic fluid through an opening in the end caps. The hydraulic pressure was controlled by an external mechanical pump, and monitored by pressure gages in the line.

c. Cylinder Testing Procedure

The following procedure was followed in performing the burst test on electroformed nickel cylinders:

(1) Inspect cylinder.

(2) Locate and bond 4 strain gages on the center line between the two cylinder ends at 90° intervals so that the strain during the test shall be measured in the hoop direction.

(3) Assemble cylinder on the bottom end cap with epoxy.

(4) Assemble top end cap on cylinder, epoxy seal and cure.

(5) Connect strain gauge leads to the strain recorder and perform all necessary balancing of the instruments with the gauges.

(6) Insert hydraulic line into end cap and slowly fill cylinder until fluid level overflows from top end cap.

(7) Center entire hydraulic burst test assembly between the platens of the large hydraulic press and close until contact is made.

(8) Apply hydraulic pressure to approximately 250 pounds of load. Record exact load and internal pressure and record strain gauge reading.

(9) Remove load and recheck strain gauges and pressure gauge for zero return.

(10) Reapply pressure to 250 pounds and record load, pressure and strain gauge values

(11) Raise load an additional 250 pounds and record load, pressure and strain gauge values.

(12) Repeat step 11 at 200 pound intervals until 2000 pounds of load is attained.

(13) Increase load intervals to 300 pounds and take appropriate readings until cylinder failure.

(14) Calculate hoop stress at failure by

$$\text{Stress} = \frac{pR}{2t}$$

where p = internal pressure at failure

R = distance from center to wall midpoint

t = average wall thickness of cylinder

(15) Plot stress-strain curve from data obtained during the test and determine cylinder modulus, yield stress and total elongation.

(16) Remove ruptured cylinder for metallographic analysis.

The details of the strain gage electronic read out and the plotted stress-strain curves for a number of the tested cylinders is presented in Appendix B. Stress-strain plots were not obtained for all the tested cylinders, due to the lack of bonding of the strain-gages on the nickel cylinders during testing.

The test results for the cylinder testing as well as for the nickel electroform tensile properties are presented in Section E.

E. RESULTS OF CYLINDER TESTING

This section contains a summary and discussion of results concerning the physical properties of standard nickel electroformed cylinders as contrasted with the dispersion strengthened and wire-wrapped nickel electroformed cylinders. The plating parameter history and sample testing results for the standard, dispersion strengthened, and wire wrapped nickel electroforms are presented in tabular form in Appendix A.

1. Standard Nickel Cylinder Test Results

Four standard nickel cylinders were fabricated and hydraulically tested to failure. Two of the cylinders were tested in the as produced condition and two in a heat treated condition. The test results are shown in Table VIII.

The standard nickel cylinders exhibited an average hoop tensile strength of 80,000 psi, a yield strength of 65,000 psi and a modulus of 25.6×10^6 psi as measured by strain gage readings during the hydraulic burst test. The test mandrel tensile specimens which were electroformed along with cylinders measured in the normal manner exhibited an average ultimate tensile strength of 102,500 psi, a yield strength of 70,000 psi and a modulus of 25.2×10^6 psi. The modulus values compare favorably on the two test geometrics and the yield strengths are comparable within experimental error, but the ultimate tensile strength of the tensile coupon is higher than the hoop tensile of the cylinder by 22,500 psi. These results indicate the difficulty of making comparisons of physical properties of a material when measured by different techniques on different test geometries.

There are a number of possible explanations for the difference between the hoop tensile strength of the cylinder and the ultimate tensile strength of the tensile coupon. First, there are differences in testing techniques, the burst testing is less accurate than the tensile testing due to problems in strain gage adhesion and plastic deformation of the cylinder before rupture. Second, there are differences in test geometry. The tensile coupons are machined from flat plates which does not involve machining on the surface to be fractured. The cylinders were always ground on the outside surface to remove protrusions and maintain a uniform wall thickness. The

TABLE VIII - Standard Nickel Cylinders

Sample No.	Test Cylinder No.	Test Cylinder Condition	Cylinder Hoop Tensile Strength x 10^3 psi	Cylinder Yield Strength 0.2% offset x 10^3 psi	Cylinder Young's Modulus x 10^6 psi	Method of Failure	Test Mandrel Tensile Strength x 10^3 psi Rt	Test Mandrel Tensile Strength x 10^3 psi 1500°F	Test Mandrel Elongtn. % Rt	Test Mandrel Elongtn. % 1500°F	Test Mandrel Young's Modulus x 10^6 psi	Test Mandrel Yield Strength x 10^3 psi
243-88PM	7	As Produced	84.1	77.0	26.0	Pin Hole Failure	112	*	10.8	*	23.2	73.2
243-93PM	8	As Produced	75.7	53.0	24.2	Rupture	93	*	5.5	*	*	*
243-100PM	10	Heat Treated	63.4	19.0	26.0	Rupture	114	8.5	9.7	6.5	28.2	74.6
243-139PM	12	Heat Treated	44.8	*	*	Rupture	94.1	6.4	10.9	2.9	24.0	62.1

*Data not measured due to insufficient material or instrumentation failure.

effect of surface grinding on the cylinder properties was not determined experimentally but would be expected to have an effect similar to annealing.

However, in spite of the differences noted above, some comparisons of physical properties improvements must be based on comparisons of strengthened material versus non strengthened material produced at the same time due to variations in nickel electroform properties depending upon the state of the bath.

The cross-section and fracture surface photomicrographs of the standard nickels cylinders are shown to illustrate the mode of failure of this material. Figure 13 shows cylinder number 8 after burst test rupture. Figure 14 shows a close-up view of the fracture surface of cylinder 8. While figure 15 shows the fracture surface area. Figure 16 shows the rupture of cylinder 10. Figures 17 and 20 show the fracture surfaces of the heat treated cylinders 10 and 12 after rupture in the burst test. Figures 18, 19, 21 and 22 show the non-fractured and fracture surfaces of cylinders 10 and 12.

2. Thoria Dispersion Strengthened Nickel Cylinder Test Results

Four thoria dispersed nickel cylinders were fabricated and two were hydraulically tested to failure. The two heat treated cylinders could not be tested due to severe oxidation in the furnace during heat treatment. This oxidation was caused by a nitrogen valve malfunction during the heat treating cycle. The test results for the as produced cylinders is shown in Table IX.

The as produced dispersion strengthened nickel cylinders showed an average hoop strength of 97,500 psi with a yield strength of 67,000 psi and a modulus of 36.4 x 10^6 psi. The test mandrel tensile specimens exhibited an average ultimate tensile strength of 92,800 psi, a yield strength of 60,000 psi and a modulus of 22.6 x 10^6 psi.

The dispersion strengthened cylinders showed an increase of 17,500 psi or 22% over the standard nickel hoop strength in the as deposited condition. Also, the tensile strength of the as produced dispersion strengthened electrodeposited nickel shows an increase of approximately 20,000 psi over conventional TD nickel sheet at room temperature. The yield strength of the dispersion strengthened electrodeposited nickel is higher by approximately 15,000 psi than the TD nickel sheet at room temperature. However, at a

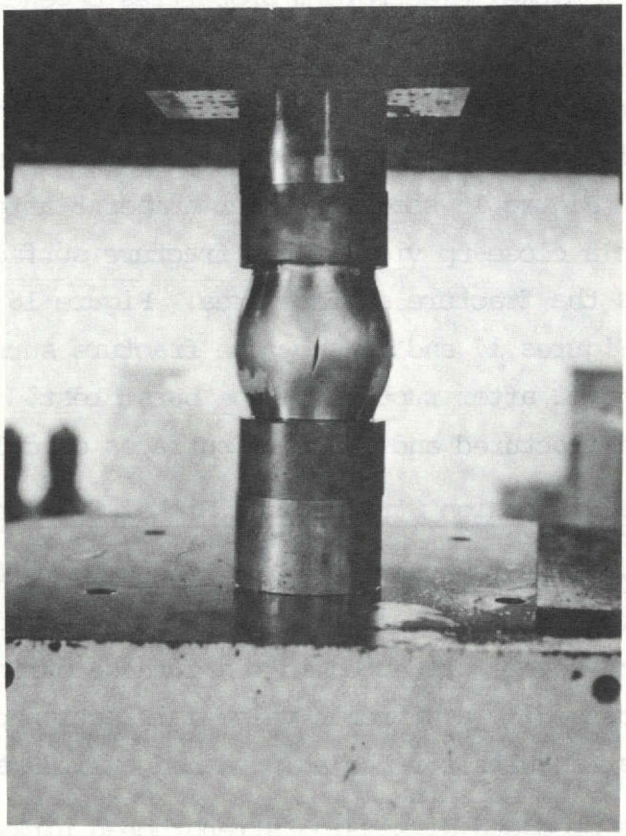


Figure 13. Standard Nickel Cylinder
After Burst Test

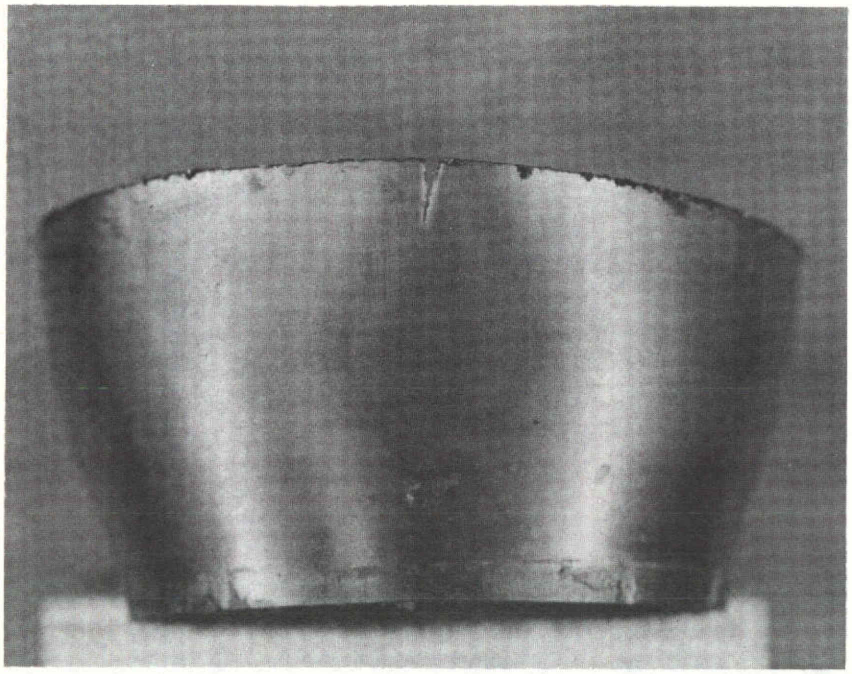


Figure 14. Fracture Surface After Burst Test of Standard Cylinder 8

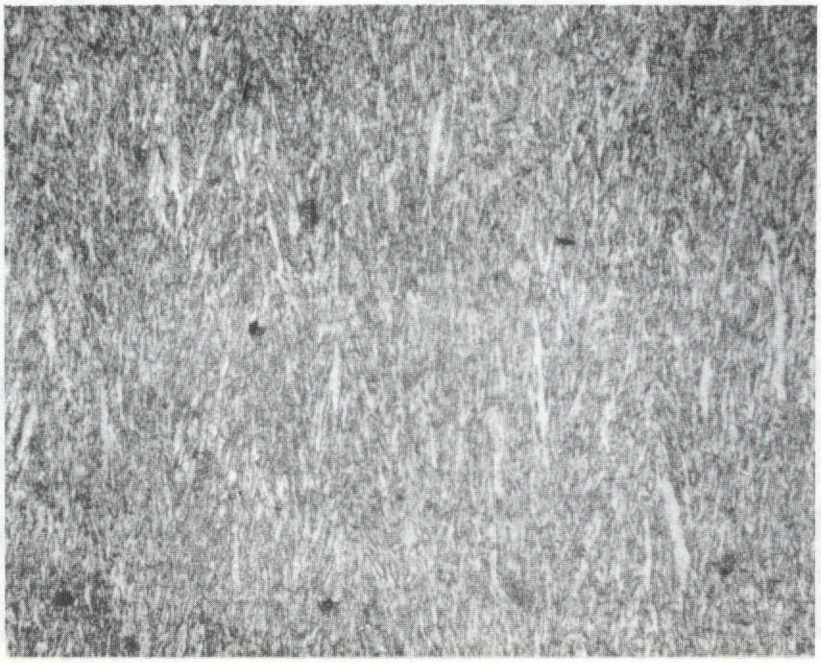


Figure 15. Fracture Surface Area of Standard Nickel Cylinder 8, 100X

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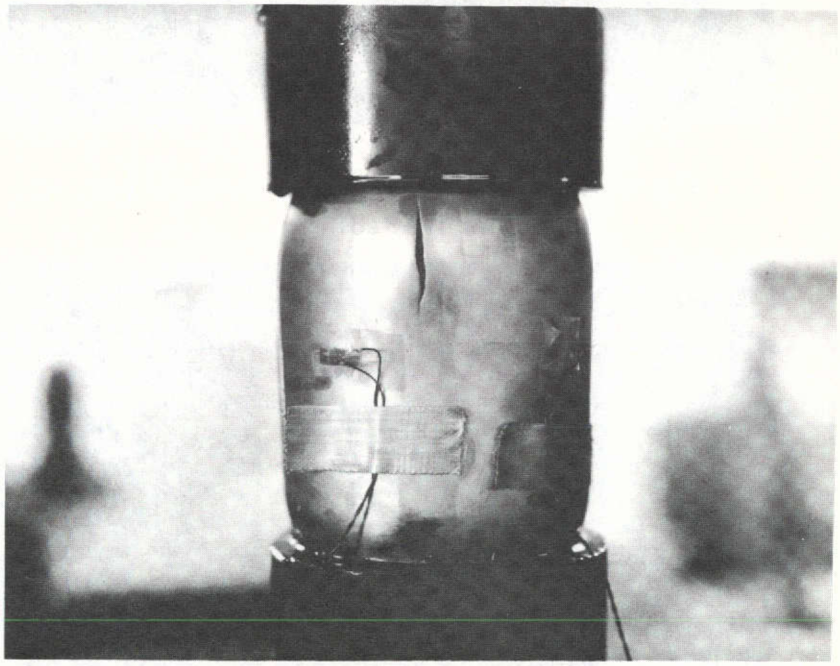

Figure 16. Fracture Surface of Heat Treated Standard Cylinder 10 After Burst

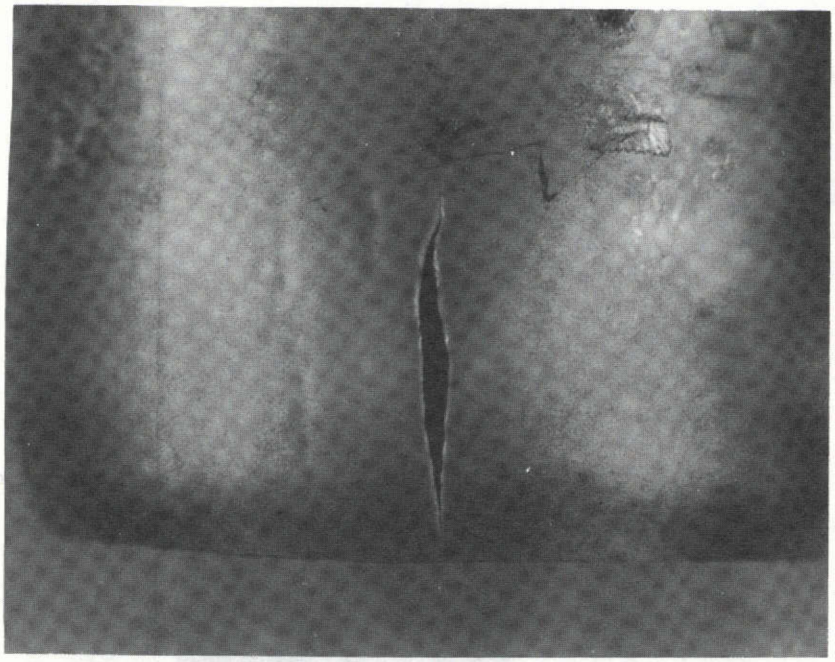

Figure 17. Fracture Surface of Cylinder No. 10
(Heat Treated)

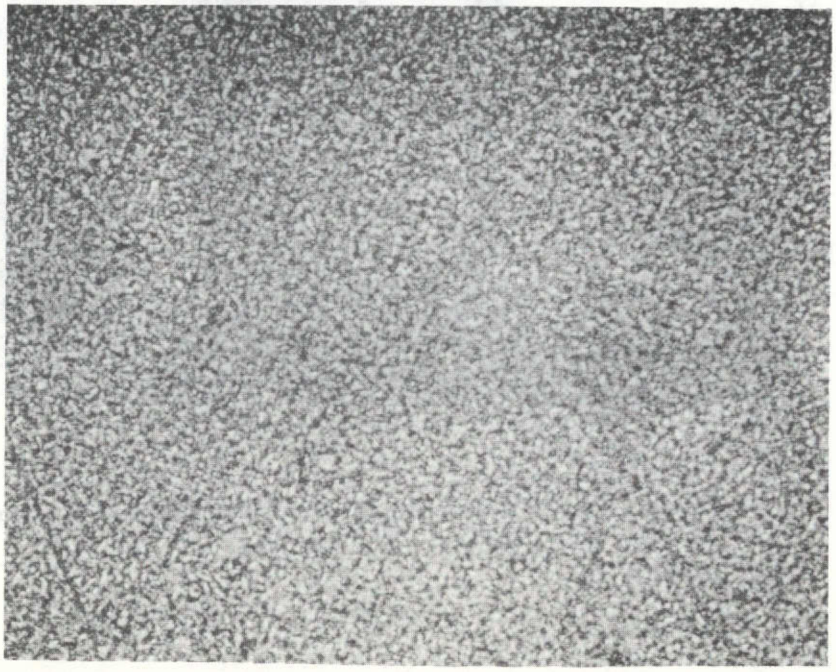

Figure 18. Cross Sectional View of Heat Treated
Standard Cylinder 10, 100X

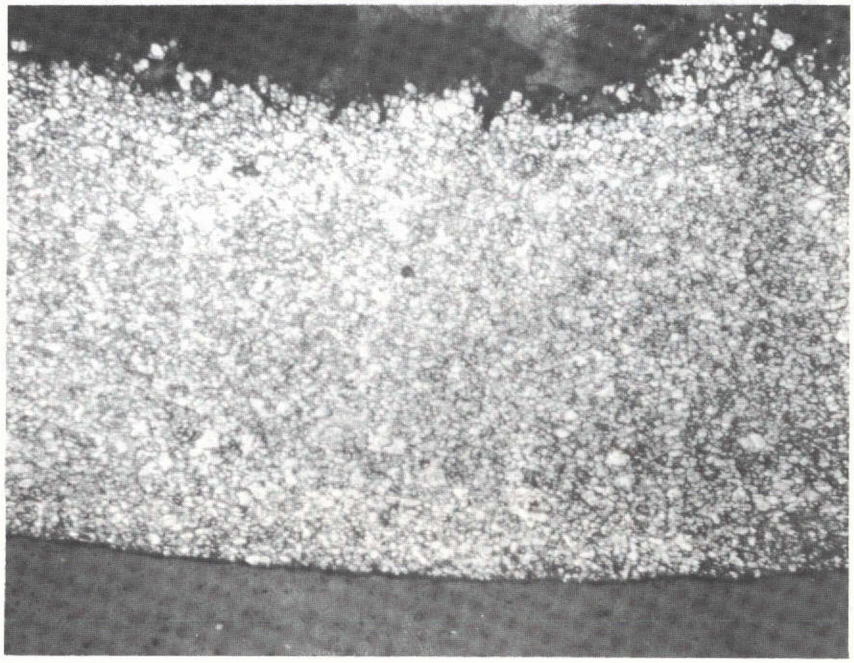

Figure 19. Fracture Section of Heat Treated Standard Cylinder 10, 100X

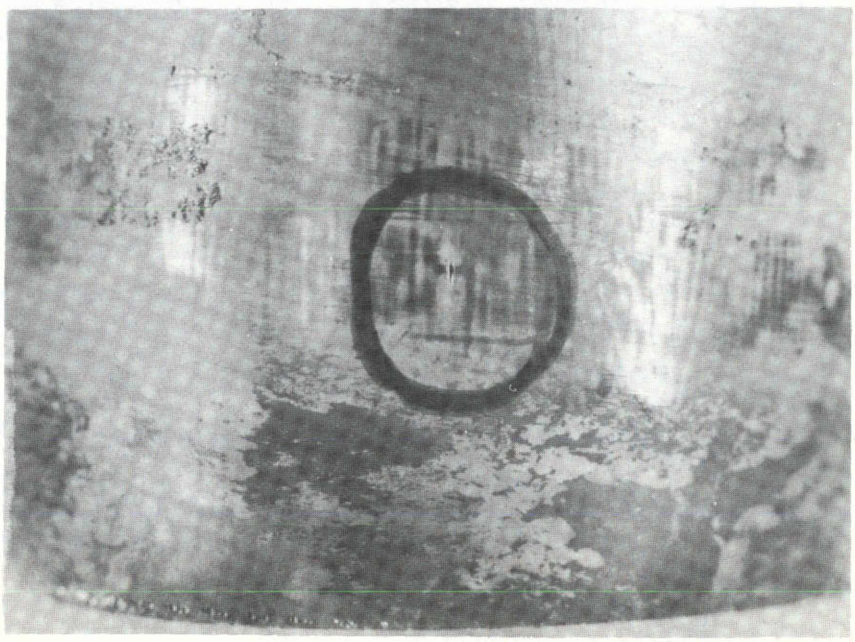

Figure 20. Fracture Surface of Cylinder No. 12 (Heat Treated)

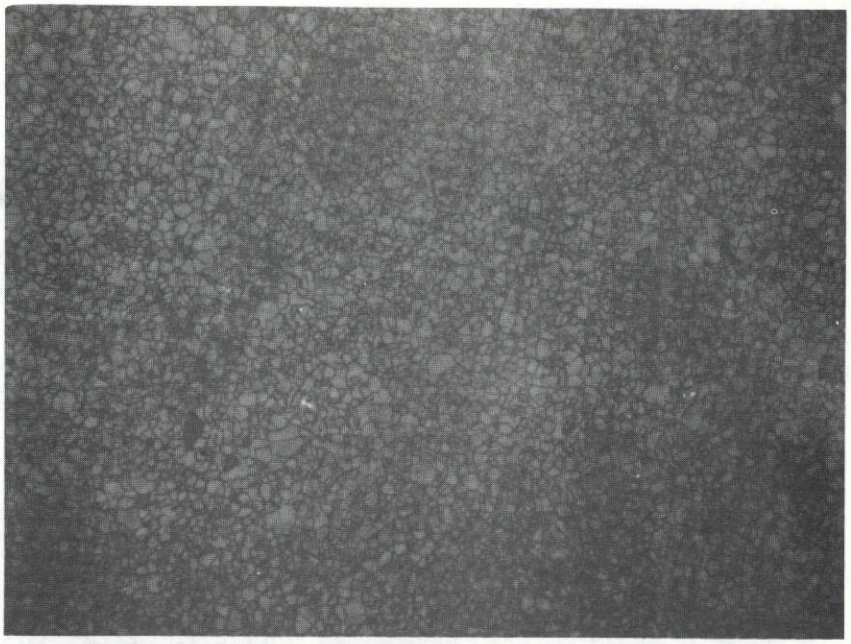

Figure 21. Cross Sectional View of Heat Treated Standard Cylinder 12, 100X

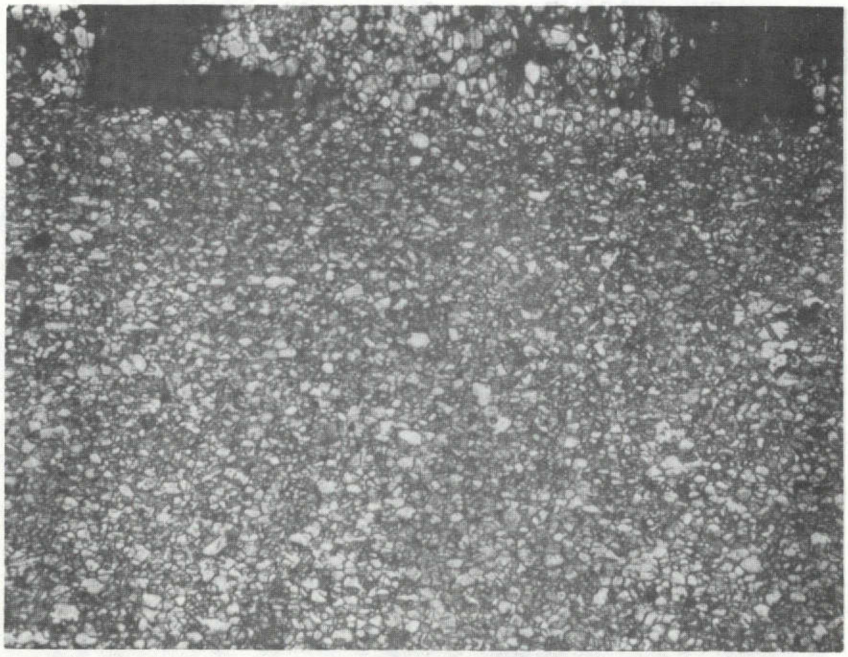

Figure 22. Fracture Section of Heat Treated Standard Cylinder 12, 100X

TABLE IX – Dispersion Strengthened Cylinders

Sample No.	Test Cylinder No.	Test Cylinder Condition	Cylinder Hoop Tensile Strength x 10^3 psi	Cylinder Yield Strength 0.2% offset x 10^3 psi	Cylinder Young's Modulus x 10^6 psi	Method of Failure	Test Mandrel Tensile Strength x 10^3 psi		Test Mandrel Elongtn. %		Test Mandrel Young's Modulus x 10^6 psi	Test Mandrel Yield Strength x 10^3 psi
							Rt	1500°F	Rt	1500°F		
246-2PM	17	As Produced	102	67	36.4	Rupture	89.9	8.1	8.6	2.0	23.0	61.3
246-3PM	18	As Produced	93	*	*	Rupture	95.7	6.7	8.4	3.7	21.2	58.7
246-6PM	20	Heat Treated	**	**	**	---	101.4	13.5	7.2	4.6	23.6	69.6
246-6PM2	21	Heat	**	**	**	---	119.0	12.1	3.5	5.5	24.9	83.3

*Data not measured due to strain gage failure.
**Cylinders not tested due to severe oxidation during heat treatment.

temperature of 1500°f, the tensile strength of the dispersion strengthened electrodeposited nickel drops to one third of the strength of TD nickel at that temperature.

Some cross-section and fracture surface photomicrographs of the dispersion strengthened nickel cylinders are shown to characterize and illustrate the mode of failure of the cylinders. Figures 23 and 24 show the fracture surface of dispersion strengthened cylinder 17 which appears to be a brittle fracture. Figures 25 through 29 show the metallurgical structure of as produced, tested and heat treated dispersion strengthened cylinders.

3. Wire Wrapped Nickel Cylinder Test Results

These test cylinders can best be analysed as a steel wire-nickel matrix composite system. In the fiber direction or hoop direction, the composite modulus and strength is principally determined by the volume ratio of the constituents (rule of mixtures). In the transverse direction the modulus and strength is principally determined by the properties of the nickel matrix alone. When the composite is stressed in the fiber or hoop direction, the matrix will yield first and, if a sufficient bond exists, share its load with the wires. The wires will undergo failure first and initiate failure for the whole composite. In the transverse direction, the transverse strength will be essentially the matrix strength applied to the whole cross section in the case of good bonding or less than the matrix strength in the case of poor bonding.

The test results for the as produced cylinders is shown in Table X, along with the test results for the mandrel test coupons. This is a case where the data must be interpreted on a comparison of strengthened material versus non-strengthened material produced at the same time. The wire wrapped cylinder hoop strengths range from 99,000 to 148,000 psi with a corresponding range for the nickel test coupons of 60,000 to 100,000 psi ultimate tensile. The wire wrapped cylinders show an increase in strength over the standard nickel test samples of 26,000 to 66,800 psi, which is a 26 to 104% increase.

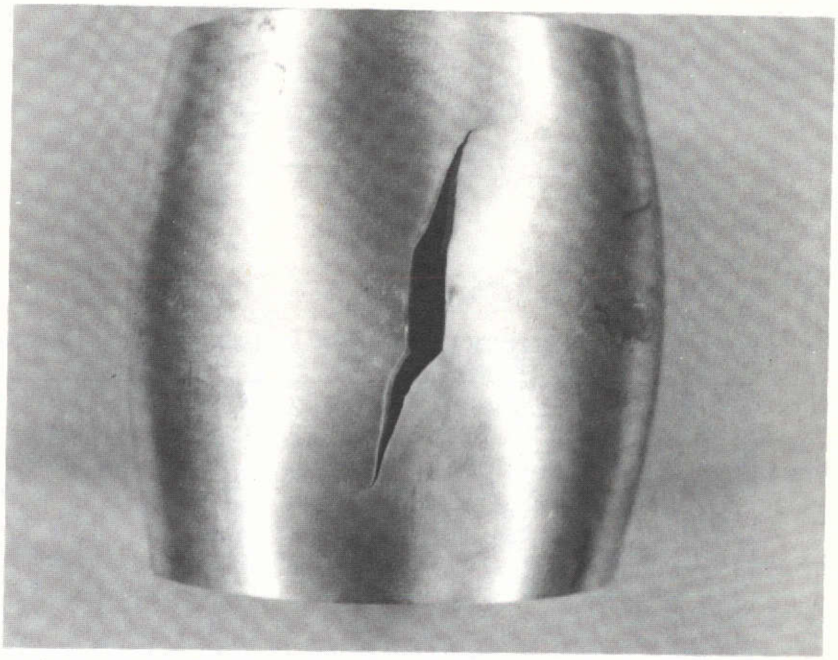

Figure 23. Fracture Surface of Dispersion Strengthened Cylinder No. 17

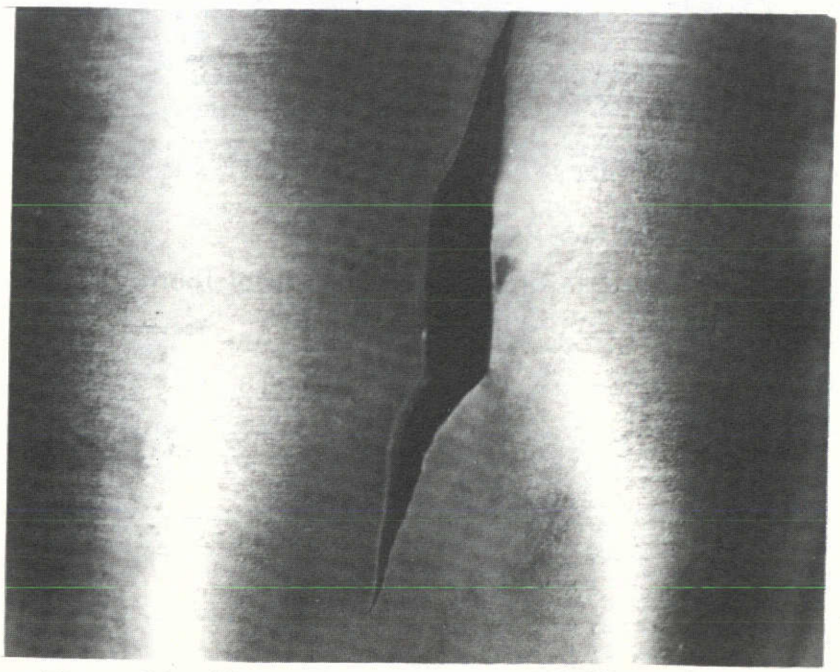

Figure 24. Close-Up of Fracture Surface of Dispersion Strengthened Cylinder No. 17

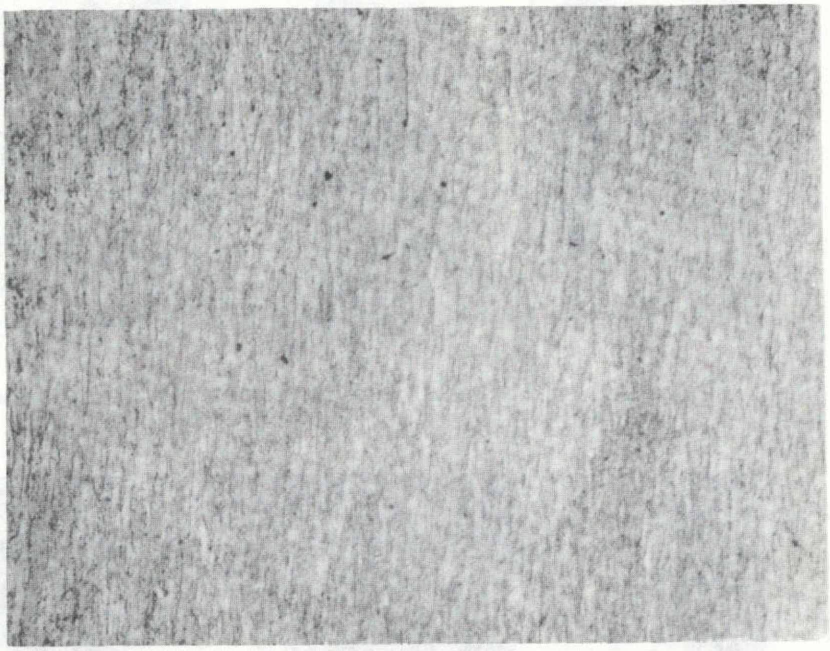

Figure 25. Cross Sectional View of Thoria Dispersed Cylinder 17, 100X

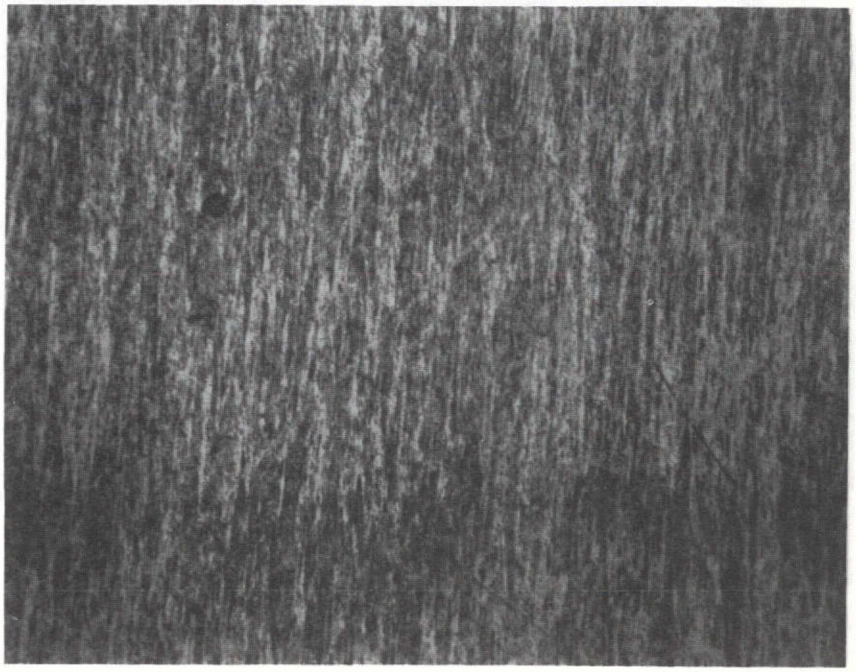

Figure 26. Cross Sectional View of Thoria Dispersed Cylinder 18, 100X

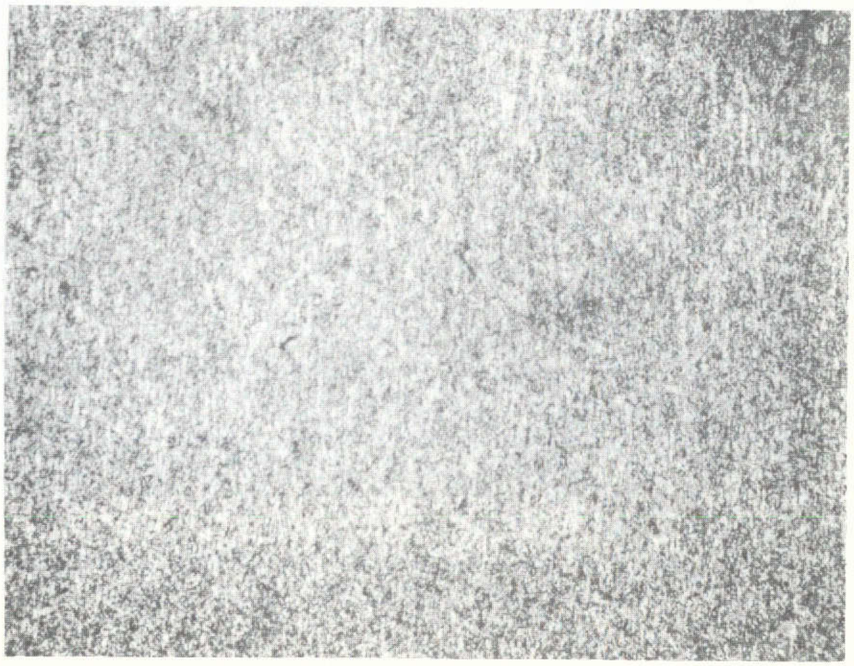

Figure 27. Fracture Section of Thoria Dispersed Cylinder 18, 100X

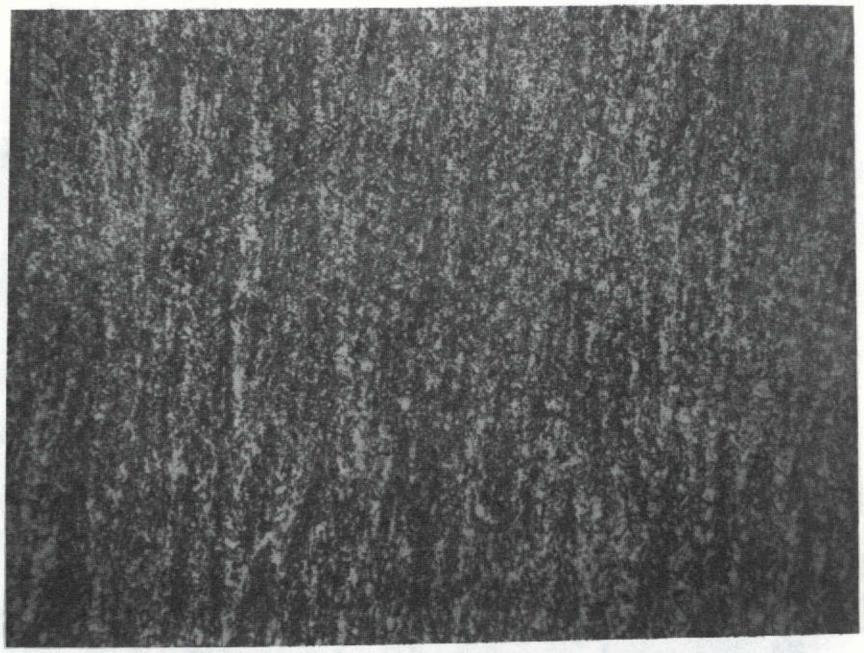

Figure 28. Cross Sectional View of Thoria Dispersed Heat Treated Cylinder 20, 100X

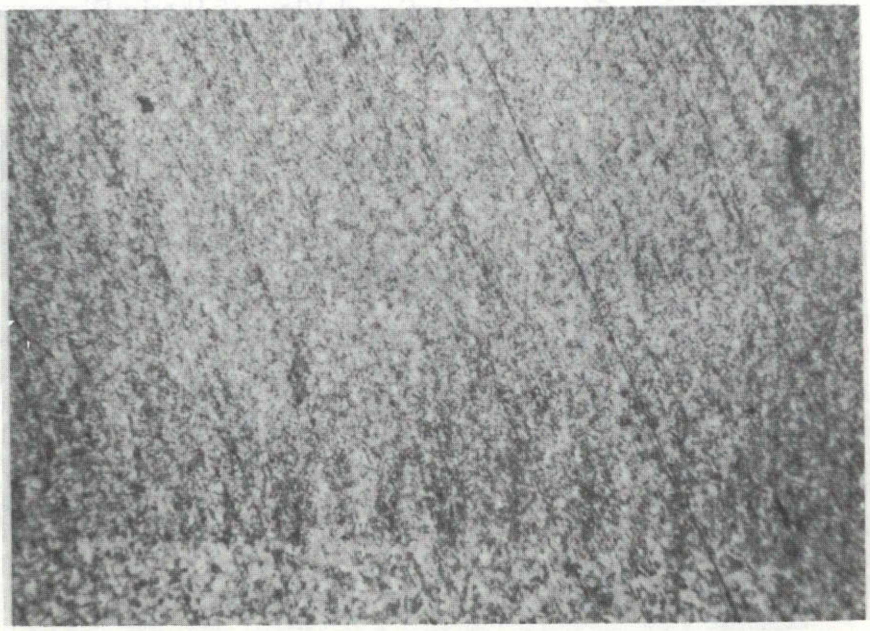

Figure 29. Cross Sectional View of Thoria Dispersed Heat Treated Cylinder 21, 100X

TABLE X – Wire Wrapped Cylinders

Sample No.	Test Cylinder No.	Test Cylinder Condition	Cylinder Hoop Tensile Strength x 10^3 psi	Cylinder Yield Strength 0.2% offset x 10^3 psi	Cylinder Young's Modulus x 10^6 psi	Method of Failure	Test Mandrel Tensile Strength x 10^3 psi Rt	Test Mandrel Tensile Strength x 10^3 psi 1500°F	Test Mandrel Elongtn. % Rt	Test Mandrel Elongtn. % 1500°F	Test Mandrel Young's Modulus x 10^6 psi	Test Mandrel Yield Strength x 10^3 psi
243-99PM	9	As Produced	126	73	29.2	Rupture	100	*	*	*	*	*
243-143PM	13	As Produced	125	82	26.0	Rupture	80.1	8.2	13.3	14.0	22.0	49.9
243-144PM	14	As Produced	99.2	58	23.2	Rupture	60.1	9.2	16.4	19.7	22.0	36.7
243-146PM	15	As Produced	131	*	*	Rupture	64.2	12.9	14.7	12.3	21.5	38.6
243-148PM	16	As Produced	**	**	**	---	74.2	13.5	13.0	10.7	20.5	48.5
243-4PM	19	As Produced	148.2	*	*	Rupture	100	*	*	*	*	*
246-7PM	22	As Produced	Not tested									
246-8PM	23	As Produced	Not tested									
246-9PM	24	As Produced	Not tested									

*Data not measured due to insufficient material or instrumentation failure.
**Cylinder not tested--sent to NASA Lewis.

The hoop tensile data shown in Table X agrees with the values calculated on the basis of volume fraction of the wire and matrix components. For cylinders 9-15, the larger diameters wire, with a tensile strength of 277,000 psi was used for fabrication. The smaller diameter wire with a tensile strength of 390,000 psi was used to fabricate cylinder 19. The comparison of calculated with measured values of hoop strength, yield strength and modulus for the wire wrapped nickel cylinders is presented in Table XI.

The volume fractions of the wire reinforcement range from 0.12 to 0.31. The measured hoop strengths agree quite well with the calculated values. This indicates that this composite system is behaving with a predictable stress-strain relationship of elastic deformation at the initial loading, then plastic deformation of the matrix with elastic deformation of fibers until composite fracture. Figures 30 through 42 show the fracture surfaces and cross-sections of several of the burst wire wrapped cylinders.

TABLE XI – Comparison of Measured Properties of Tested Cylinders with Calculated Rule of Mixture Properties

Test Cylinder No.	Calculated Wire Volume Fraction	Calculated Composite Hoop Tensile Strength $\times 10^3$ psi	Measured Composite Hoop Tensile Strength $\times 10^3$ psi	Calculated* Composite Yield Strength $\times 10^3$ psi	Measured Composite Yield Strength 0.2% offset $\times 10^3$ psi	Calculated** Composite Young's Modulus $\times 10^6$ psi	Measured Composite Young's Modulus $\times 10^6$ psi
9	0.14	124.8	126	--	73	22.8	29.2
13	0.30	139.2	125	109.9	82	23.8	26.0
14	0.17	96.9	99.2	73.0	58	23.1	23.2
15	0.31	130.2	131	--	--	--	--
19	0.12	134.8	148.2	--	--	--	--

*Yield strength of steel wire assumed to be 250,000 psi
**Modulus of steel wire assumed to be 28.0×10^6 psi.

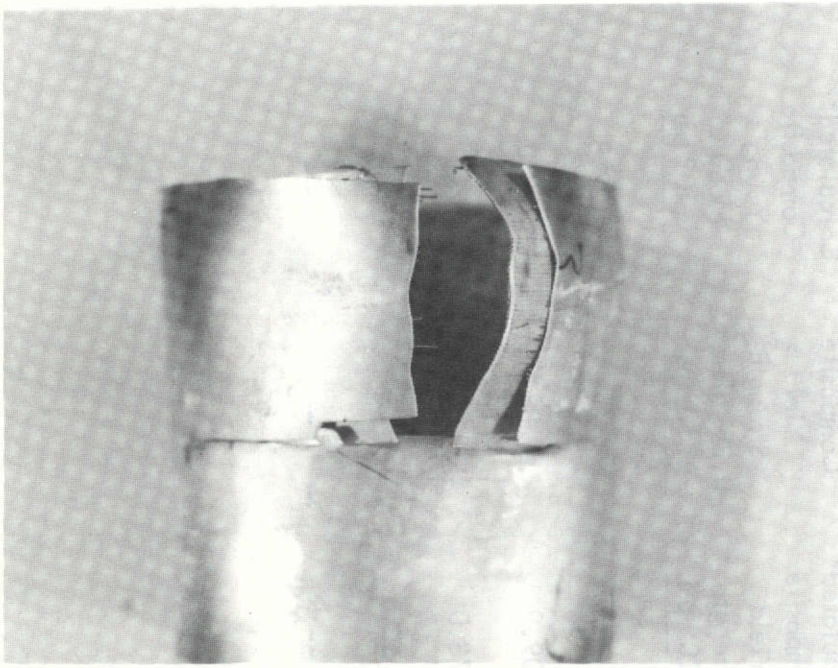

Figure 30. Fracture Surface of Wire Wrapped Cylinder No. 9 Showing Layer Delamination After Burst

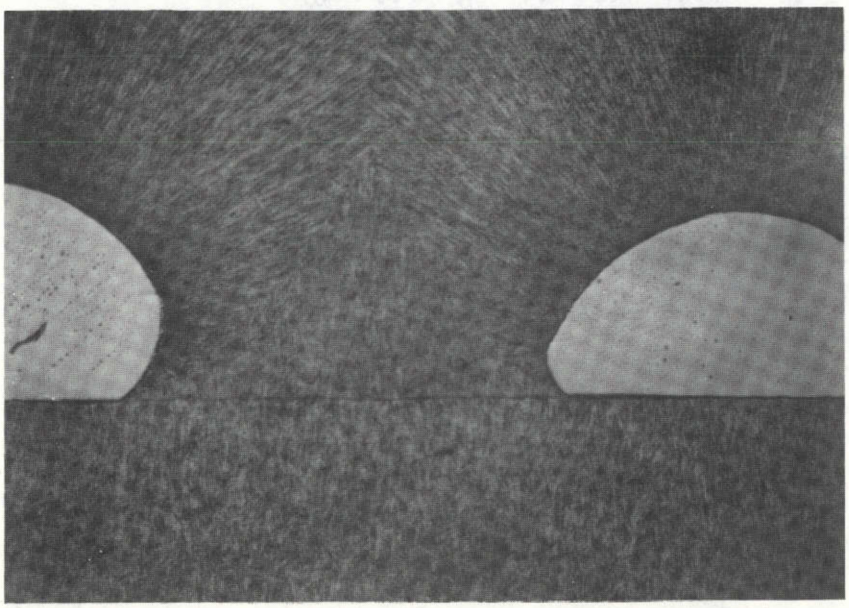

Figure 31. Cross Sectional View of 20 Mil Wire Wrapped Cylinder 9, 100X

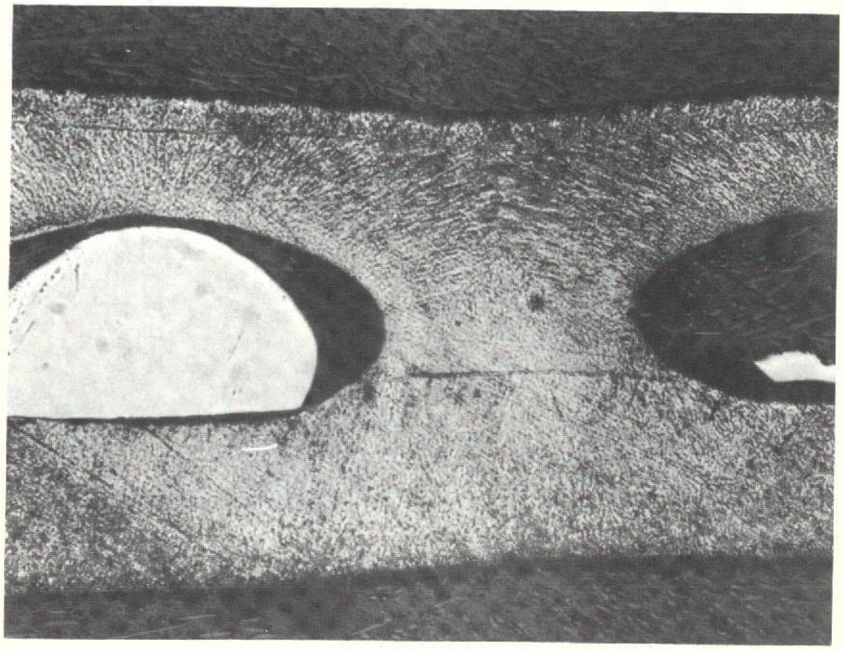

Figure 32. Fracture Section of Wire Wrapped Cylinder 9, 100X

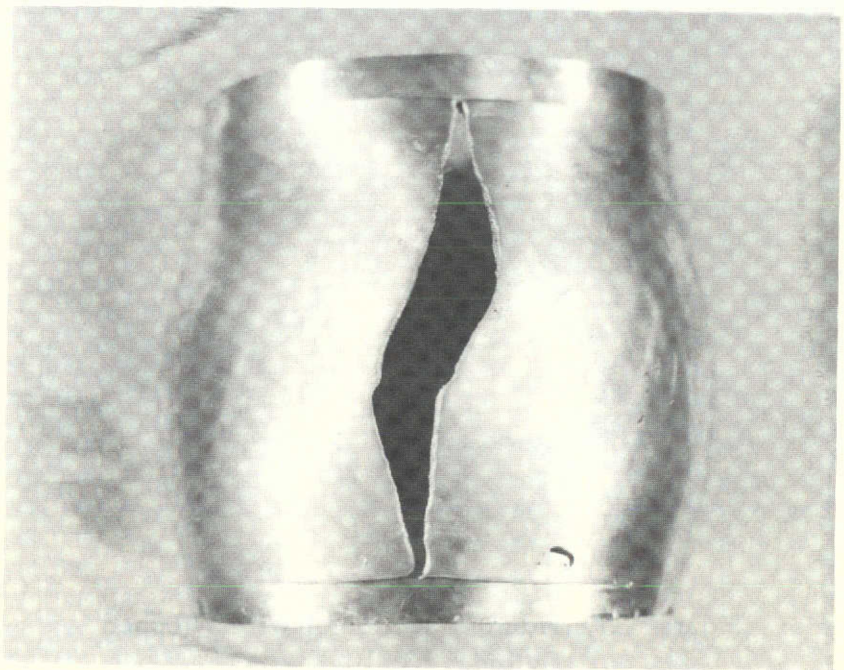

Figure 33. Fracture Surface of Wire Wrapped Cylinder No. 13

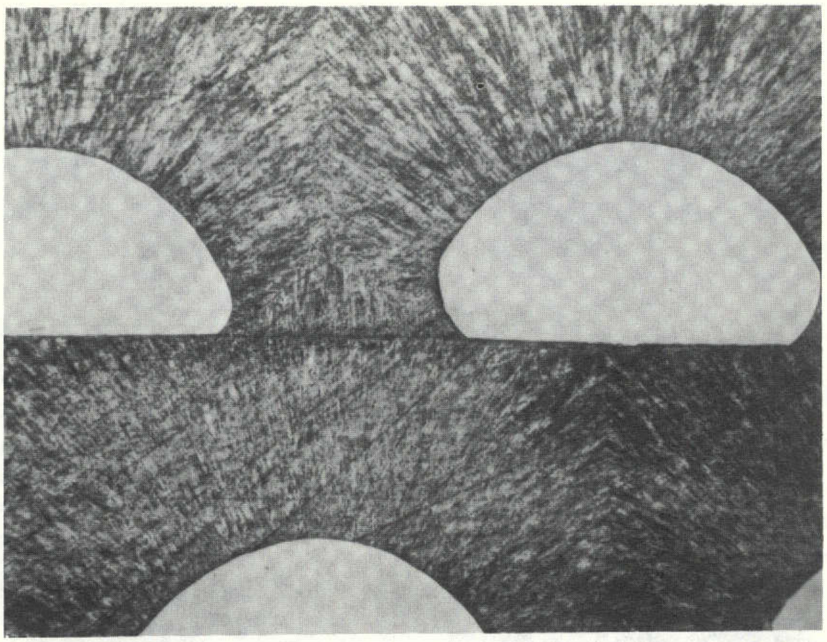

Figure 34. Cross Sectional View of Wire Wrapped Cylinder 13, 100X

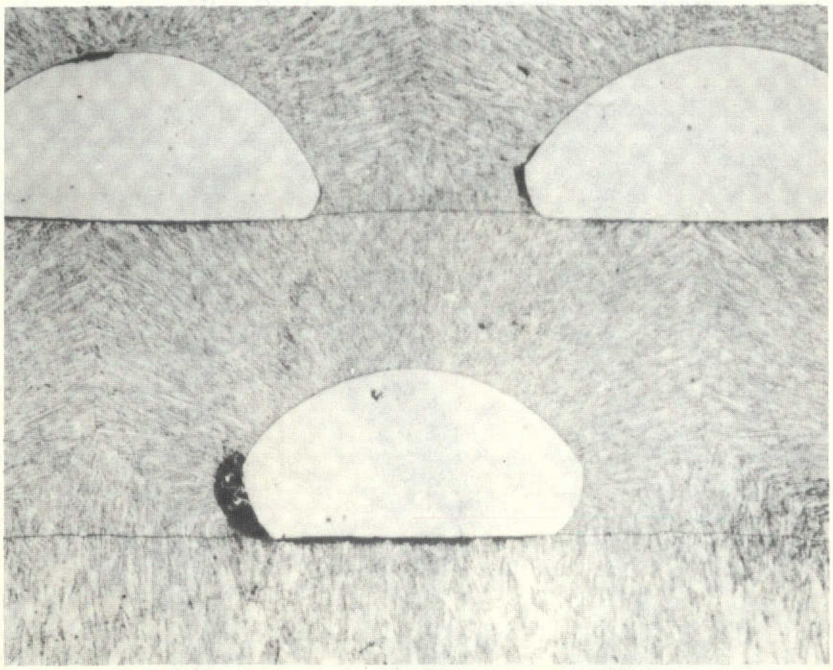

Figure 35. Fracture Section of Wire Wrapped Cylinder 13, 100X

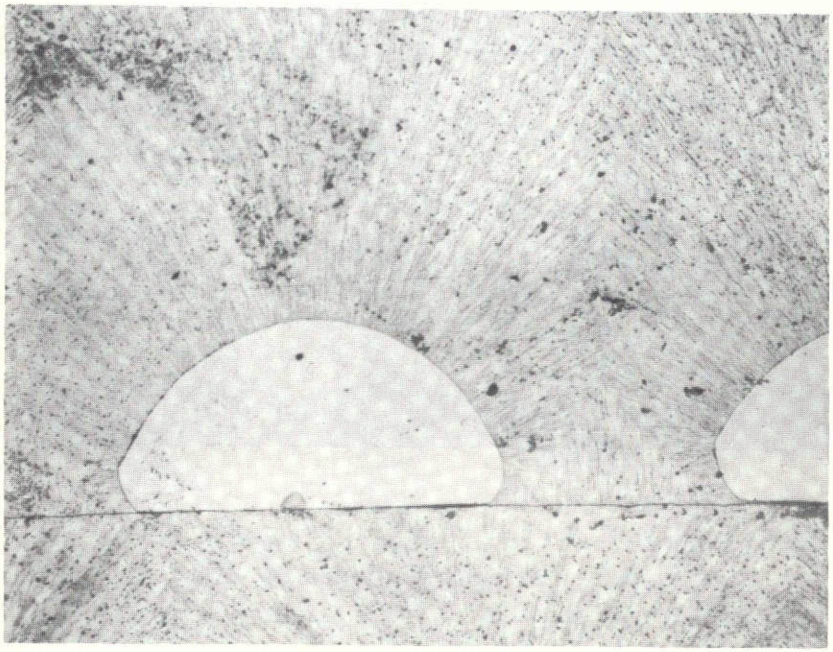

Figure 36. Cross Sectional View of Wire Wrapped Cylinder 15, 100X

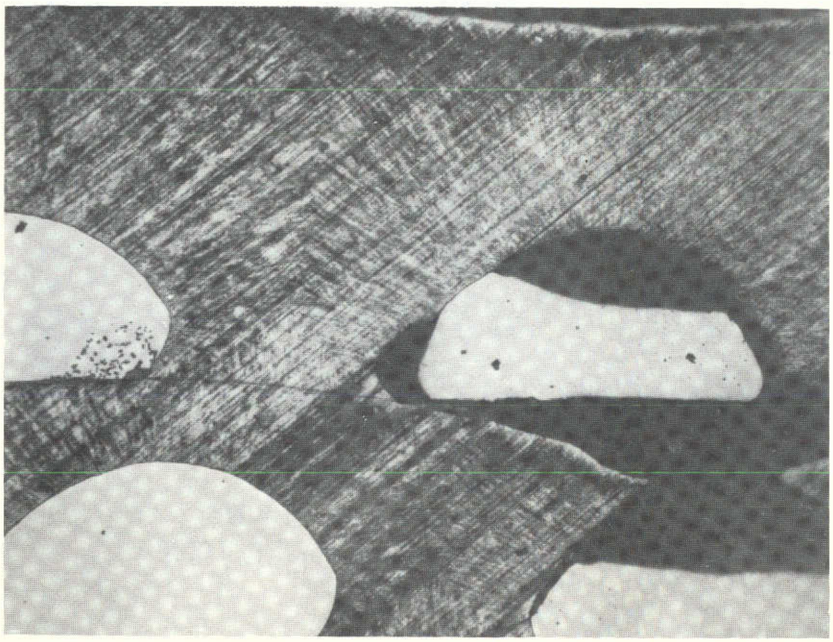

Figure 37. Fracture Section of Wire Wrapped Cylinder 15, 100X

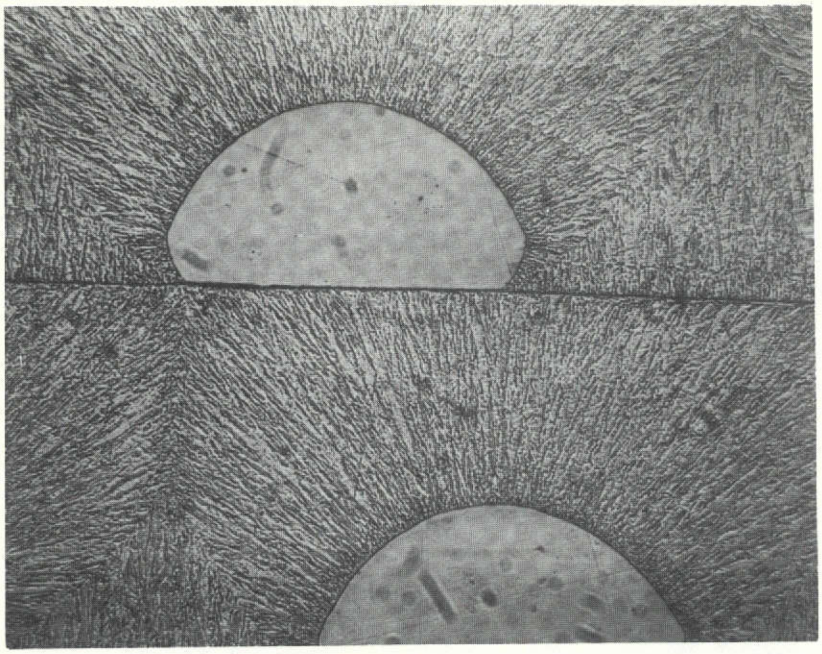

Figure 38. Cross Sectional View of Wire Wrapped Cylinder 14, 100X

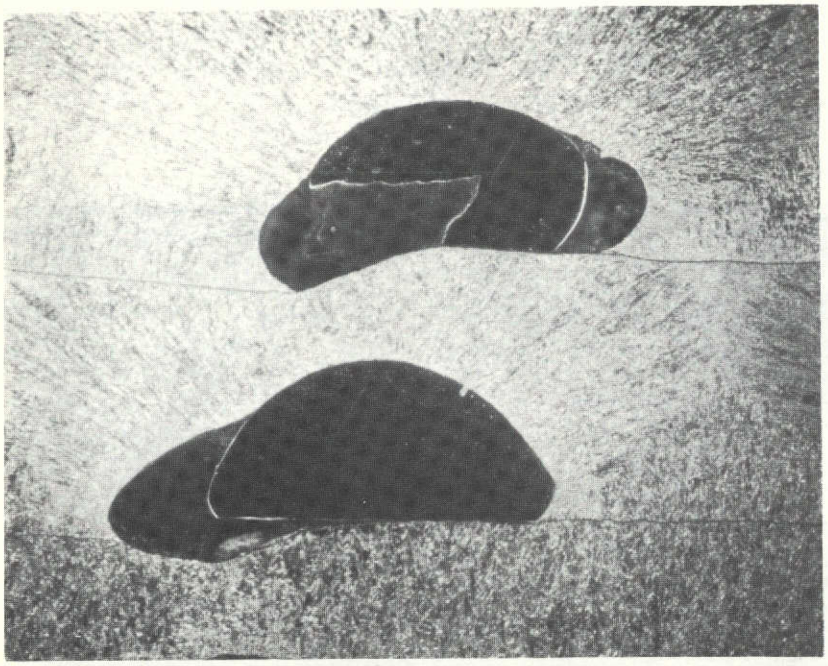

Figure 39. Fracture Section of Wire Wrapped Cylinder 14, 100X

GTC 297-40

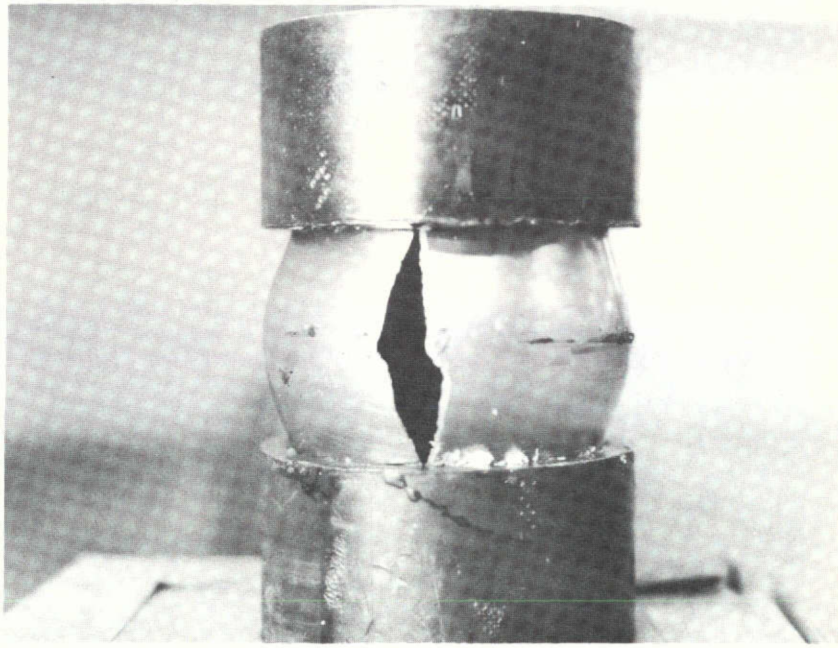

Figure 40. Fracture Surface of Wire Wrapped Cylinder 19

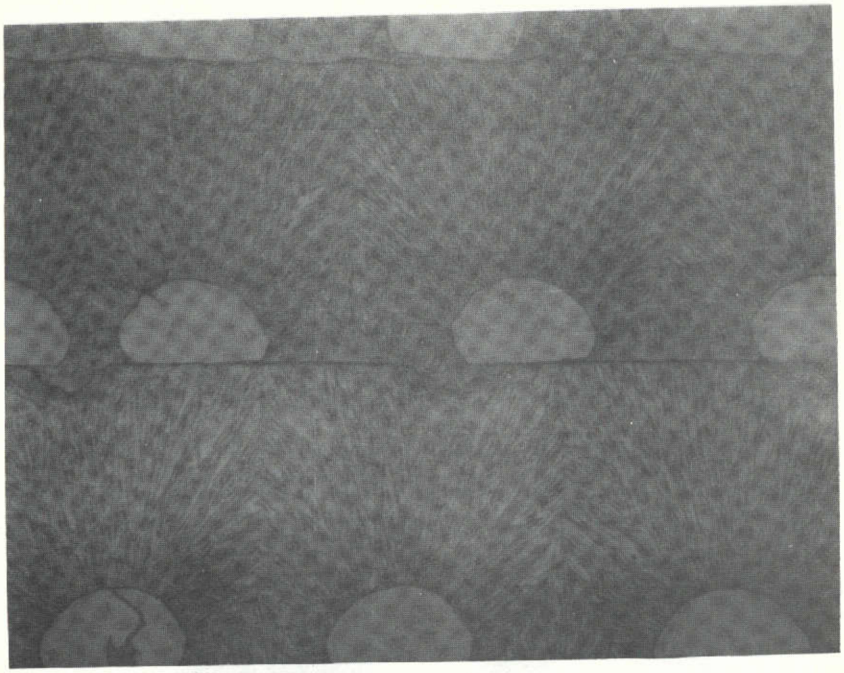

Figure 41. Cross Sectional View of Wire Wrapped Cylinder 19, 100X

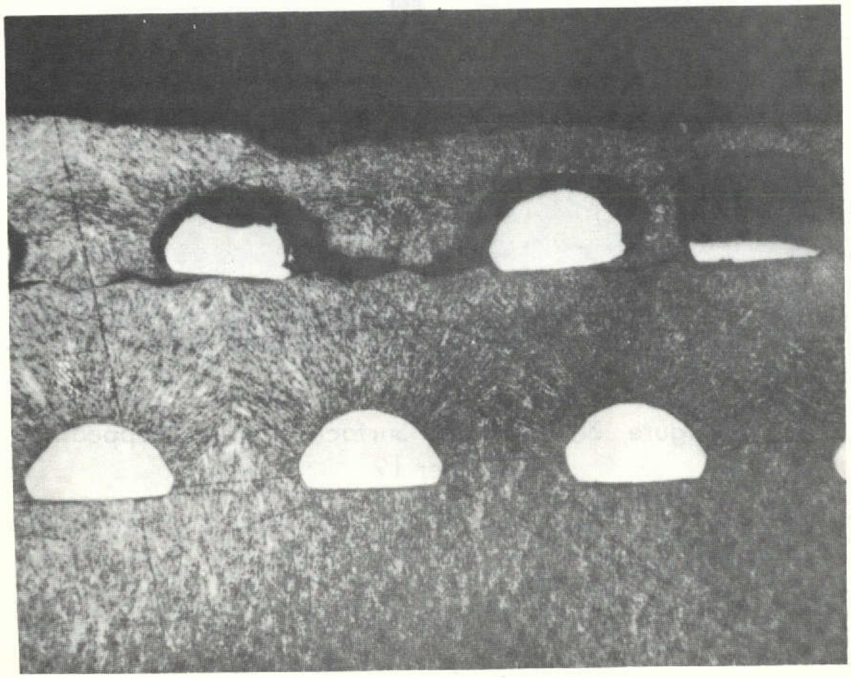

Figure 42. Fracture Section of Wire Wrapped Cylinder 19, 100X

IV - SUMMARY OF RESULTS

The objective of this program was to advance and improve the electroforming process by developing techniques to reinforce or strengthen the deposited material. This effort was accomplished by researching and evaluating methods to produce both dispersion strengthened and wire reinforced nickel cylindrical structures.

The initial task was to verify the physical properties of an electroformed nickel from a sulfamate-chloride bath formulation with target properties of 100,000 psi and 10% elongation over a two inch gage section. These target properties were verified using 50 to 70 mil thick test plates electroformed in the bath. However, numerous test samples produced from this bath as shown in Appendix A, indicate that there is a delicate balance of properties with regard to tensile strength and two inch elongation results. The target properties of 100,000 psi and 10% are achievable and have been achieved, however if the tensile strength decreased to below 85,000 psi, then a corresponding increase above 10% occurs in the elongation value. This same phenomenon occurs when the tensile strength increases above 115,000 psi and the elongation correspondingly decreases below 10%. The metallurgical explanation for this phenomenon has not been delineated at this time. However, the phenomenon is real enough and experimentally verified.

During the course of this program, it was found to be difficult to consistently achieve the target physical properties with the bath. Thus some comparisons of physical property improvements must be based upon comparisons of strengthened material versus non strengthened material produced at the same time. Both the dispersion strengthening technique and the wire reinforcing technique produced electroformed nickel cylinders which exhibited higher strengths than the standard electroformed nickel.

The standard nickel cylinders exhibited an average hoop strength of 80,000 psi, a yield strength of 65,000 psi and a modulus of 25.6×10^6 psi. The as produced dispersion strengthened nickel showed an average hoop strength of 97,000 psi with a yield strength of 67,000 psi. This is an increase of 17,000 psi or 21% over the standard nickel hoop tensile strength. The wire wrapped cylinders showed an increase in strength over the standard nickel test samples of 26,000 to 66,800 psi which is in the range of 26 to 104% increase over the base standard nickel. These latter test results are indicative of a volume percent wire reinforcement ranging from 15 to 31. The fabrication with higher volume percent reinforcement should show even greater strength increases. The measured hoop strengths agree with calculated composite strengths based on rule of mixtures.

The fracture surfaces of the burst cylinders indicate a ductile hoop failure mode with the possible exception of the dispersion strengthened cylinder number 17 which may have undergone brittle fracture. In all cases cylinder elongation of greater than 5% was observed at failure. The fracture of the wire wrapped cylinders is indicative of a brittle fiber - ductile matrix composite.

SECTION V - REFERENCES

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SECTION VI

APPENDIX A

CUMULATIVE EXPERIMENTAL DATA FOR NICKEL ELECTROFORMING

Table I. Cumulative Experimental Data For Nickel Electroforming

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Coating Thickness (Mils)	Disposition	Average Tensile Strength Kpsi	Average Elongation % (2 in. Gage)	General Remarks
PSN-1	243-2	40	4.1	48	16.0	-	-	Not Tested	-	-	Dummy Plate - Pitted
PSN-1	243-3a	20	4.2	50	15.75	-	-	Not Tested	-	-	Dummy Plate - Pitted
PSN-1	243-3b	40	4.2	50	21.5	412	17	Not Tested	-	-	Dummy Plate - Pitted
PSN-1	243-3c	40	4.1	50	17	-	-	Not Tested	-	-	Dummy Plate - Pitted
PSN-1	243-4a	30	4.1	52	67.5	1316	-	Not Tested	-	-	Dummy Plate - Pitted
PSN-1	243-4b	40	4.1	53	22.25	530	19	Tested	114.3	9.0	Somewhat Pitted
PSN-1	243-5	40	4.1	52	24.25	529	19	Not Tested	-	-	Somewhat Pitted
PSN-1	243-6a	50	4.0	52	16.5	617	19	Tested	131.8	8.4	Somewhat Pitted
PSN-1	243-6b	30	3.9	50	46.0	780	-	Not Tested	-	-	Dummy Plate
PSN-1	243-7a	50	3.8	52	15.75	606	22	Tested	119.5	7.9	
PSN-1	243-7b	40	3.9	53	*-	-	-	Not Tested	-	-	*Lost Electrical Connection
PSN-1	243-8a	50	3.9	52	21.0	628	21	Tested	88.0	9.6	
PSN-1	243-8b	50	3.9	52	24.5	690	22	Tested	98.0	9.5	
PSN-1	243-10	50	**	50	22.0	637	22	Tested	92.0	11.0	**PH Meter Broken
PSN-1	243-11A	60	**	52	20.8	727	26	Not Tested	-	-	Pitted
PSN-1	243-11B	60	**	51	*-	2736	80	Tested	95.0	10.7	*Lost Electrical Connection
PSN-1	243-12	60	**	54	21.0	687	24	Tested	88.0	9.6	
PSN-1	243-13A	40	4.0	49	54.5	1503	46	Tested	91.3	13.0	

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Coating Thickness (Mils)	Disposition	Average Tensile Strength Kpsi	Average Elongation % (2 in. Gage	General Remarks
PSN-1	243-13B	40	4.1	52	23.6	510	17	Tested	90.8	12.3	
PSN-1	243-14A	40	4.0	51	42.4	885	27	Tested	96.6	13.4	
PSN-1	243-15A	40	4.2	51	23.25	544	19	Not Tested	-	-	Somewhat Pitted
PSN-1	243-15B	60	4.1	52	111.25	3582	96	Tested	83.3	14.4	
PSN-1	243-17	40	4.0	53	66	1782	-	Not Tested	-	-	Pitted
PSN-1	243-19A	40	4.0	53	16.5	409	-	Not Tested	-	-	Pitted
PSN-1	243-19B	40	4.0	53	15.5	435	-	Not Tested	-	-	Pitted
PSN-1	243-20	40	4.1	54	72	1610	-	Not Tested	-	-	Pitted
PSN-1	243-21	40	4.0	51	66	1957	54	Tested	98.9	10.9	Verification Sample
PSN-1	243-22	40	4.0	52	*-	1403	40	Tested	94.4	11.5	*Lost Electrical Connection
PSN-1	243-23 (Cylinder)	40	4.0	52	*-	5162	90	Not Tested	-	-	Pitted on Bottom Section - Electrical Power Failure

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Coating Thickness (Mils)	Disposition	Average Tensile Strength Kpsi	Average Elongation 2 in. Gage %	General Remarks
PSN-Cl-1	243-25 (Cylinder)	40	4.0	53	71	3498	76	Tested	91.9	10.6	Samples taken from test coupon mandrel
ESN-Cl-2	243-28E	40	4.1	48	21	210	27.5	Tested	90.3	6.4	
PSN-Cl-1											Loss of bath--pump failure
PSN-Cl-2	243-29	40	4.4	52	23	570	--	Not Tested	--	--	Pitted
ESN-Cl-1	243-29E	40	4.1	50	18	180	--	Not Tested	--	--	Pitted
PSN-Cl-2	243-30	40	4.4	52	--	--	--	Not Tested	--	--	Lost electrical connections
PSN-Cl-2	243-31	40	4.4	51	87	1729	42	Tested	119.3	7.6	
ESN-Cl-1	243-31E-1	40	4.2	50	95	1900	69	Tested	94.8	11.1	
ESN-Cl-1	243-31E-2	40	4.1	54	--	--	--	Not Tested	--	--	
PSN-Cl-2	243-32	50	4.3	52	--	--	--	Not Tested	--	--	
ESN-Cl-1	243-32E	40	4.1	52	6	60	--	Not Tested	--	--	Pitted
PSN-Cl-2	243-33P	40	4.15	52	20	504	--	Not Tested	--	--	Pitted
ESN-Cl-1	243-33E	40	4.4 Anode 4.15 Cathode	51	21	210	--	Not Tested	--	--	Pitted
PSN-Cl-2	243-34P	40	4.1	51	89.5	1759	46	Tested	106.5	9.8	
ESN-Cl-1	243-35E	40	4.35 Anode 4.1 Cathode	49	22.5	225	--	Not Tested	--	--	Pitted

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Coating Thickness (Mils)	Disposition	Average Tensile Strength Kpsi	Average Elongation 2 in. Gage %	General Remarks
ESN-Cl-1	243-36E-1	40	4.1 Anode 4.1 Cathode	50	19	190	--	Not Tested	--	--	Pitted
ESN-Cl-1	243-36E-2	40	4.2 Anode 4.1 Cathode	50	26	260	--	Not Tested	--	--	Fewer pits
ESN-Cl-1	243-38E	40	4.1 Anode 4.1 Cathode	51	69	690	--	Pitted	--	--	Pitted
PSN-Cl-2	243-38P	40	4.0	52	--	--	52	Tested	109.7	10.9	
PSN-Cl-2	243-39P	40	4.05	52	43	1047	31.5	Tested	107.3	10.2	
ESN-Cl-1	243-40E	40	4.1 Anode 4.1 Cathode	51	21	210	--	Not Tested	--	--	Pitted
ESN-Cl-1	243-41E	40	4.05 Anode 4.1 Cathode	51	21	210	--	Not Tested	--	--	Pitted
PSN-Cl-2	243-42P (Cylinder)	40	4.05	52	74	3884	74	Tested	72.3	13.8	Samples taken from test coupon mandrel

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Coating Thickness (Mils)	Disposition	Average Tensile Strength Kpsi	Average Elongation 2 in. Gage	General Remarks
E-SNCl-1	243-44E	40	4.5A 3.8C	52	--	--	--	Not Tested	--	--	Metallic contamination in bath
E-SNCl-2	243-47E	40	4.2A 4.2C	48	94	940	--	Not Tested	--	--	Pitted
P-SNCl-2	243-47P	40	4.0	51	24	612	--	Not Tested	--	--	Filters clogged
E-SNCl-2	243-49E	40	4.25A 4.25C	50	23	230	--	Not Tested	--	--	Pitted
E-SNCl-2	243-50E	40	4.05A 4.05C	51	25	250	--	Not Tested	--	--	Agitation off during run
P-SNCl-2	243-51P	40	4.05	51	22	444	--	Not Tested	--	--	
P-SNCl-2	243-51P2	40	4.1	51	16.5	923	--	Not Tested	--	--	Full depth double test plates — pitted one side
E-SNCl-2	243-52E	40	4.1A 4.2C	50	47	470	--	Not Tested	--	--	Surface inclusions
P-SNCl-2	243-52P	40	4.1	50	15	976	--	Not Tested	--	--	Double test plates — pitted one side
E-SNCl-2	243-53E	40	4.1A 4.1C	51	92	920	75	Tested	76.5	14.1	
P-SNCl-2	243-54P	40	4.1	51	67	3497	--	Not Tested	--	--	Double test plates — pitted one side
E-SNCl-2	243-55E	40	4.3A 4.3C	50	42	420	--	Not Tested	--	--	

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Thickness (Mils)	Disposition	Average Tensile Strength Kpsi	Average Elongation 2 in. Gage	General Remarks
PSN-Cl-2	243-55P	40	4.3	51	19	369	--	Not Tested	--	--	Pitted — low boric acid
PSN-Cl-2	243-56P	40	4.2	51	68	1694	--	Not Tested	--	--	Double test plates — top front pitted
ESN-Cl-2	243-56E	40	4.1A 4.1C	51	18	180	--	Not Tested	--	--	Slightly pitted
ESN-Cl-2	243-57E	40	4.1A 4.1C	51	24	240	27	Tested	87.5	9.1	Slightly pitted
ESN-Cl-2	243-59E	40	4.1A 4.1C	49	42	420	--	Not Tested	--	--	Slightly pitted
PSN-Cl-2	243-60P	23	4.1	52	--	965	--	Not Tested	--	--	Lost electrical contact
PSN-Cl-2	243-62P	24	4.0	50	68	2220	49 top 43 bottom	Tested	99.4 top 104.6 bot.	12.1 top 9.8 bot.	Double test plates
ESN-Cl-3	243-63E	20	4.0A 4.0C	49	72	1440	--	Not Tested	--	--	Pitted
ESN-Cl-2	243-64E	20	4.2A 4.4C	50	19	380	--	Not Tested	--	--	Pitted
ESN-Cl-2	243-65E	20	4.2A 4.3C	49	6	120	--	Not Tested	--	--	Started to pit so removed
ESN-Cl-2	243-65E2	20	4.2A 4.3C	49	--	--	--	Not Tested	--	--	
ESN-Cl-3	243-66E	20	4.6A 4.2C	49	66	330	--	Not Tested	--	--	Some small pits
PSN-Cl-2	243-67P	23	4.15	50	16	488	--	Not Tested	--	--	Double test plates — low on antipit agent

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Thickness (Mils)	Disposition	Average Room Temp. Tensile Strength Kpsi	Average Elongation 2 in. Gage	Young's Modulus 10^6 psi	General Remarks
ESN-Cl-2	243-67E	40	4.1A 4.1C	50	18	180	--	Not Tested	--	--	--	
ESN-Cl-2	243-69E2	20	4.0A 4.0C	50	68	340	33.5	Tested	103.3	11.5	--	
ESN-Cl-3	243-69E3	20	4.1A 4.1C	50	68	340	--	Not Tested	--	--	--	Pitted — agitation off during run
ESN-Cl-3	243-70E3	20	4.3A 4.3C	49	43	215	--	Tested	105	5.9	--	Test samples tore out of grips - sample too thin
ESN-Cl-2	243-70E2	20	4.1A 4.1C	49	24	120	--	Not Tested	--	--	--	
ESN-CL-2	243-71E2	20	4.0A 4.0C	49	24	120	--	Not Tested	--	--	--	
ESN-Cl-3	243-72E3	20	4.25A 4.25C	49	8	48	--	Not Tested Cross sectional	--	--	--	Slightly pitted lower edge
ESN-Cl-2	243-73E2	25	4.1A 4.1C	49	96.5	194	--	Not Tested	--	--	--	Wire reinforced sample
ESN-Cl-3	243-73E3	20	4.25A 4.25C	49	96	470	--	Not Tested	--	--	--	
ESN-Cl-2	243-76E2	20	3.9A 4.0C	50	70	350	37	Tested	129.3	6.5	27.2	Aluminum oxide dispersion
PSN-Cl-2	243-76P2	25	4.2	49	--	--	--	Not Tested	--	--	--	
PSN-Cl-2	243-77P2M (Cylinder)#5	20	4.1	49	44	1246	92	Tested	116	10.5	24.1	Taken out early - mandrel pitting - lower test mandrel ok.

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Thickness (Mils)	Disposition	Average Room Temp. Tensile Strength Kpsi	Average Elongation 2 in. Gage	Young's Modulus 10^6 psi	General Remarks
PSN-Cl-2	243-79P2	5	4.2	49	14	--	--	Not Tested	--	--	--	Dummy plate
PSN-Cl-2	243-80P2M (Cylinder)	20	4.2	49	77	2297	75	Tested	111.7	10.8	--	Cylinder No. 6
ESN-Cl-3	243-81E3	20	4.4A	49	73	383		Testing Incomplete				
PSN-Cl-2	243-85P2	5	4.2	49	120	--	--	Not Tested	--	--	--	Dummy plate
ESN-Cl-3	243-85E3	10	5.0A 4.0C	45A 49C	120	--	--	Not Tested	--	--	--	Dummy plate
ESN-Cl-2	243-86E2	20	5.5A 4.5C	49	106	530	92	Tested	97.5	6.4	25.1	Al_2O_3 dispersion
PSN-Cl-2	243-88P2 (Cylinder)	20	4.2	49	91	2407	71	Tested	112	10.8	--	Cylinder No. 7
ESN-Cl-2	243-90E2	5	4.5A 4.05C	49	96	--	--	Not Tested	--	--	--	Dummy plate
ESN-Cl-3	243-91E3	5	3.1A 4.1C	43A 49C	8	--	--	Not Tested	--	--	--	Dummy plate
ESN-Cl-3	243-92E3	20	4.0C 3.1A	44A 49C	72	360	--	Tested	138	7.3	--	Slightly pitted on lower edge
ESN-Cl-2	243-92E2	40	3.8A 4.0C	44C	72	360	--	Tested	81.4	10.1	--	Rough plate Al_2O_3 dispersion
ESN-Cl-3	243-93E3	5	3.9A 4.25C	44A 49C	72	--	--	Not Tested	--	--	--	Dummy plate

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Thickness (Mils)	Disposition	Average Room Temp. Tensile Strength Kpsi	Average Elongation 2 in. Gage	Young's Modulus 10^6 psi	General Remarks
ESN-CI-2	243-93E2	5	4.0C 4.3A	39A 49C	96	--	--	Not Tested	--	--	--	Dummy plate
PSN-CI-2	243-93P2M (Cylinder)#8	20	3.95	49	87	2333	90 (Cylinder)	Tested	93.9	5.5	--	Test mandrel is thin due to poor contact
ESN-CI-2	243-97E2	20	4.4A 4.5C	38A 49C	100	500	98	Tested	111.2	4.5	20.7	Al_2O_3 dispersion 10 g/l bath conc.
PSN-CI-2	243-99P2M (Cylinder)	20	4.05	49	81	2200	81	Not Tested	--	--	--	Cylinder No. 9
PSN-CI-2	243-100P2M (Cylinder)	20	4.05	49	75	2101	71	Tested	114	9.7		Cylinder No. 10
ESN-CI-3	243-100E3	20	4.3A 4.25C	39A 49C	98	490	34	Tested	124	3.9	23.5	Thorium oxide dispersion
PSN-CI-2	243-99PM (Restart)	20	4.1	49	24.5	695	22	Not Tested	--	--	--	Restart of 99PM wound with "D shaped wire
ESN-CI-3	243-106E3	30	4.8A 4.0C	44A 51C	64	480	41	Tested	130.3	4.1	29.7	ThO_2 dispersion
ESN-CI-2	243-108E2	20	4.8A 4.0C	36A 49C	64	320	43	Tested	114	9.7		Al_2O_3 dispersion 16 g/l bath conc.
ESN-CI-3	243-108E3	40	4.3A 4.25C	44A 49C	64	640	54	Tested	127	3.4		ThO_2 dispersion 5 g/l bath conc.

Table I. Cumulative Experimental Data For Nickel Electrofroming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Thickness (Mils)	Disposition	Average Room Temp. Tensile Strength Kpsi	Average Elongation 2 in. Gage	Young's Modulus 10^6 psi	General Remarks
ESN-Cl-2	243-113E2	20	4.5A 4.05C	36A 50C	96	480	47	Tested	117	1.1		Al_2O_3 dispersion 25 g/l bath conc.
ESN-Cl-3	243-116E3	20	4.3A 4.2C	43A 49C	120	600	60	Not Tested				ThO_2 dispersion 5 g/l bath can.
ESN-Cl-2	243-120E2	40	4.85A 4.0C	36A 49C	160	720	61	Tested	105	2.0		Al_2O_3 dispersion 24 g/l bath conc.
ESN-Cl-2	243-126E2	60	4.7A 4.1C	35A 50C	47	705	69	Tested	84.7	6.3		Al_2O_3 dispersion 24 g/l bath conc.
ESN-Cl-3	243-127E3	50	4.2A 4.0C	39A 46C	45.5	569	59	Tested	107	5.2		ThO_2 dispersion 5 g/l bath conc.
ESN-Cl-2	243-128E2	30	4.7A 4.2C	46A 50C	95	643	79	Tested	95.0	2.9	25.3	Al_2O_3 dispersion 24 g/l bath conc.
ESN-Cl-3	243-129E3	20	4.8A 4.2C	46A 49C	70.5	353	35	Tested	129	3.4	28.8	ThO_2 dispersion 5 g/l bath conc.
PSN-Cl-2	243-136P2M (Cylinder)#11	20	4.05	49	10	740	20	Not Tested				Wire wrapped cylinder using 20 mil wire with 10 mil spacing
PSN-Cl-3	243-139PM (Cylinder) #12	20	4.1	49	62	1750	56	Tested	94.1	10.9	24.0	Standard cylinder.

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hours	Thickness (Mils)	Disposition	Average Room Temp. Tensile Strength Kpsi	Average Elongation 2 in. Gage	Young's Modulus 10^6 psi	General Remarks
PSN-Cl-2	243-143PM (Cylinder) #13	20	4.2	49	56	2100	62	Tested	80.1	13.3	22.0	Wire wrapped using 20 mil wire with 10 mil spacing.
PSN-Cl-2	243-144PM (Cylinder) #14	20	4.21	49	97	2300	62.5	Tested	60.1	16.4	22.0	Wire wrapped using 20 mil wire with 20 mil spacing
PSN-Cl-2	243-146PM (Cylinder) #15	20	4.2	49	123	2460	61.0	Tested	64.2	14.7	21.5	Wire wrapped using 20 mil wire with 10 mil spacing.
PSN-Cl-2	243-148PM (Cylinder) #16	20	4.1	49	177	3540	70	Sent to NASA Lewis	74.2	13.0	20.5	Wire wrapped using 8 mil wire with 8 mil spacing
PSN-Cl-3	246-2PM (Cylinder) #17	20	3.9	48	79	1580	70	Tested	89.9	8.6	23.0	ThO_2 Dispersion
PSN-Cl-3	246-3PM (Cylinder) #18	20	4.1	48	81	1620	71	Tested	95.7	8.4	21.2	ThO_2 Dispersion
PSN-Cl-2	246-4PM (Cylinder) #19	20	4.1	49	254.5	5090	70	Not Tested	- -	- -	- -	Wire wrapped using 8 mil wire with 8 mil spacing

Table I. Cumulative Experimental Data For Nickel Electroforming (continued)

Bath No.	Specimen No.	Current Density (asf)	pH	Temp. (°C)	Plating Time (Hrs)	Plating Amp-Hrs	Thickness (Mils)	Disposition	Average Room Temp. Tensile Strength Kpsi	Average Elongation 2 in. Gage	Young's Modulus 10 psi	General Remarks
PSN-Cl-3	246-6PM (Cylinder) #20	20	4.1	48	72	1872	68	Tested	101.4	7.2	23.6	ThO_2 Dispersion
PSN-Cl-3	246-6PMZ (Cylinder) #21	20	4.1	48	67	1742	70	Tested	119.0	3.5	24.9	ThO_2 Dispersion
PSN-Cl-2	246-7PM (Cylinder) #22	20	4.1	49	Not Finished		-	Not Tested	- -	- -	- -	Wire wrapped using 20 mil wire with 10 mil spacing 4 layers
PSN-Cl-3	246-8PM (Cylinder) #23	20	4.1	48	Not Finished		-	Not Tested	- -	- -	- -	Wire wrapped using 20 mil wire and ThO_2 dispersion
PSN-Cl-2	246-9PM (Cylinder) #24	20	4.1	49	Not Finished		-	Not Tested	- -	- -	- -	Wire wrapped using 8 mil wire with 8 mil spacing 3 layers

SECTION VI

APPENDIX B

PHYSICAL PROPERTY TESTING INSTRUMENTATION AND DATA FOR CYLINDER TESTS

A. HYDRAULIC BURST TESTING OF CYLINDERS

1. Non Constrained End Test Apparatus

This apparatus consisted of an internally pressurized hydraulic cell, with the cylinder to be tested mounted inside between a top and bottom closure. The closures were designed with gasket and "O" ring seals designed to seal to the inside surface of the cylinder. The end closures are mechanically attached to a connecting outer sleeve by steel pins. This end closure connecting sleeve serves both to retain the end closures during the test and to protect personnel and equipment from a rapid pressure release on burst. The hydraulic pressure was applied on a moveable piston mounted through the top closure by the Tinius Olsen Electromatic Testing Machine. The applied load on the piston was measured on the calibrated dial of the testing machine. This apparatus is shown schematically in Figure 43 and operational in Figure 44. This apparatus worked well when checked out using a steel cylinder machined from a solid bar of type 1116 free machining carbon steel. However, if the test cylinder is sufficiently ductile like electroformed nickel, the cylinder wall deforms and the hydraulic fluid can leak past the end closure seals. The test worked well for the carbon steel cylinder because it exhibited brittle failure, but was not successful in bursting to failure a nickel electroform cylinder.

2. Strain Gage Test Instrumentation

The strain gage electronics consisted of a standard D. C. Wheatstone bridge circuit with a self compensating strain gage in one arm of the bridge. There is a separate circuit for each of the four strain gages. Various values of shunt resistors are switched across the galvonometers to give a range of ocillograph sensitivities. At each sensitivity the

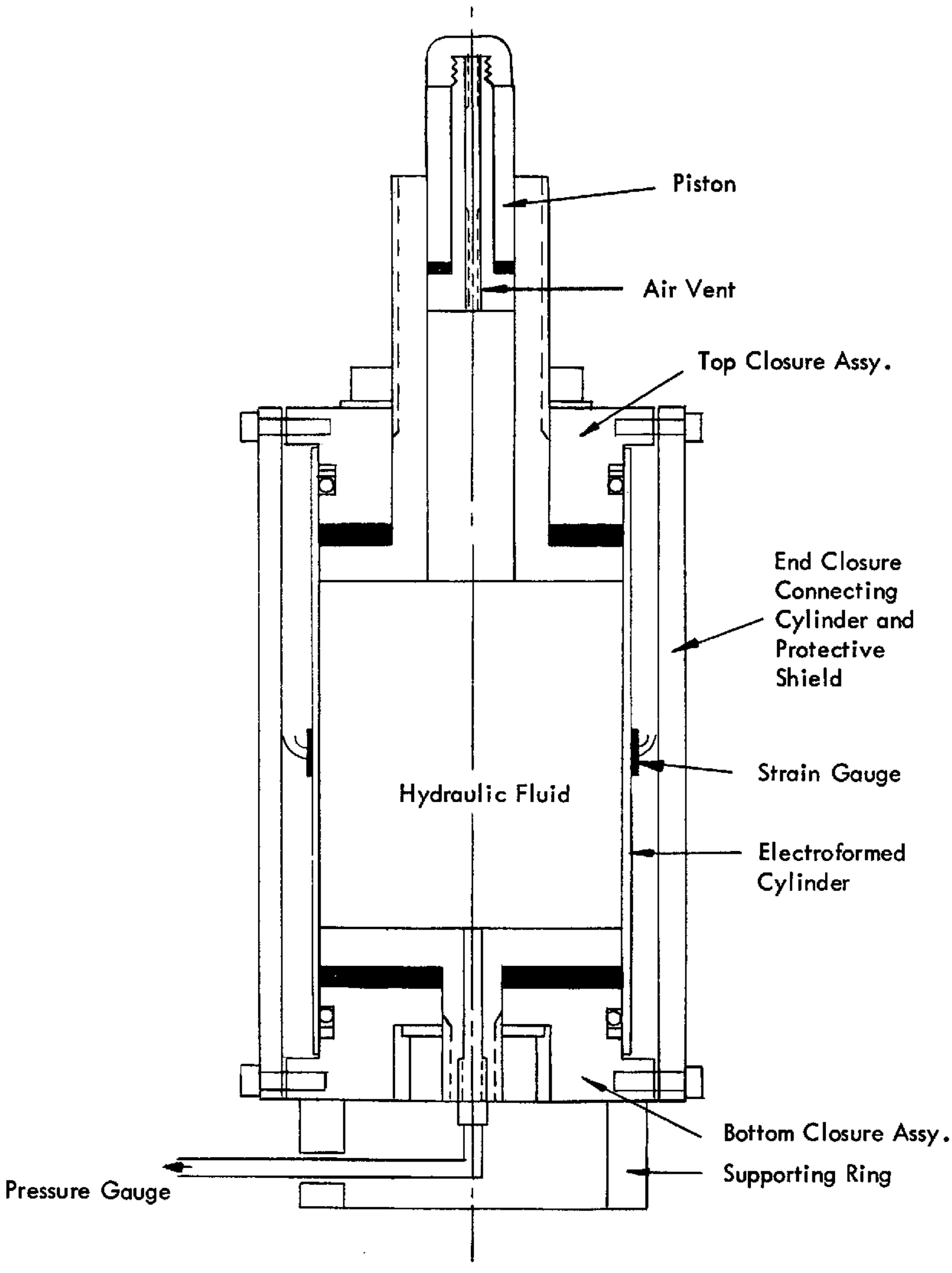

Figure 43. Schematic of Hydraulic Burst Test Assembly

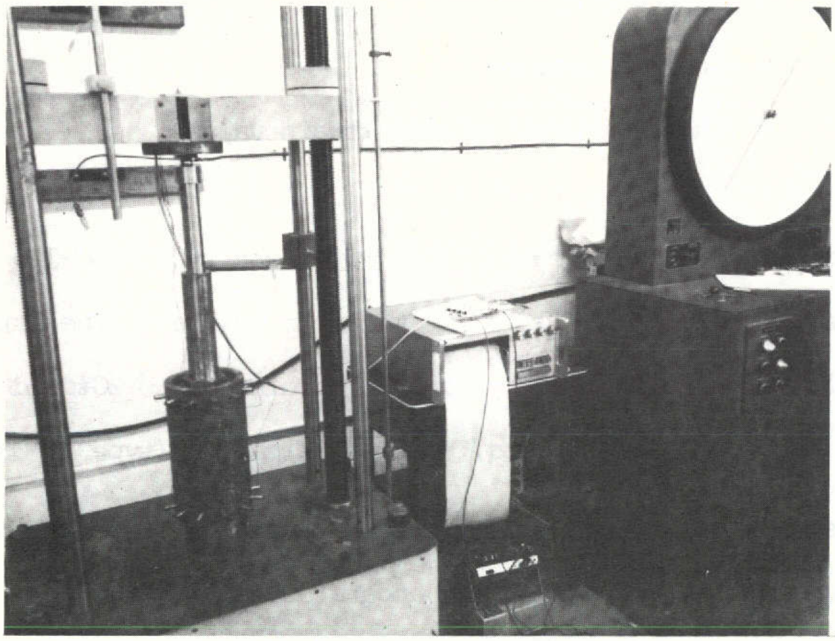

Figure 44. Hydraulic Burst Test Assembly Mounted on Tinius Olsen Testing Machine with Strain Gage Readout Equipment

galvonometer deflection is calibrated by shunting the gage with a precision resistor whose value is determined from the following formula:

$$R_{cal} = \frac{R_g}{G.F. \times \varepsilon} - R_g$$

Where R_{cal} = A resistor, which, when connected in parallel with the strain gage produces a bridge unbalance simulating a desired strain.

G.F. = Gage factor

R_g = Gage resistance

ε = The desired simulated strain value.

A Honeywell Model 1508 Visicorder fitted with four type M200-120 galvonometers is used to record the output of the four bridges. The strain gages are post yield gages, capable of measuring strains up to 20% and are manufactured by Micro Measurements, Romulus, Michigan (type EP-08-250 BG-120).

The instrumentation was checked out using a steel cylinder mounted in the non-constrained end burst test apparatus.

The specimen for this test consisted of an as-machined type 1116 steel cylinder having the following mechanical properties (literature values):

Tensile strength	-	$60\text{-}100 \times 10^3$ psi
Young's Modulus	-	30×10^6 psi
Yield Strength	-	$50\text{-}90 \times 10^3$ psi
Elongation	-	10 to 20%

The test cylinder was machined from solid stock to the following dimensions:

Length	-	8.0 inches
I.D.	-	4.0 inches
Wall thickness	-	0.070 inch.

Four strain gages were mounted on the outer surface of the cylinder at the mid-point of the cylinder length in positions around the circumference at 90° intervals. The strain sensitivity was in the hoop direction. The gages were mounted with an epoxy resin (type Ae-15) formulated by Micro Measurements for post yield gages. The test fixture with cylinder in place was set-up on the Tinius Olsen test machine and filled with a water-oil mixture as the hydraulic ram until failure of the cylinder occurred. The load at failure was used to calculate the hoop strength as follows:

$$S = \frac{DL}{2tA}$$

Where
- D = inside diameter of cylinder
- L = ultimate load on testing machine
- t = wall thickness of cylinder
- A = cross-sectional area of test piston

A cross head motion detector on the Tinius Olsen was used to relate the load applied with time and the oscillograph plotted the strain with time. The stress and strain at given times was calculated and a stress-strain curve was plotted. For this test, two bridge sentivities of 6,000 micro-strain per inch and 30,000 micro-strain per inch of chart were used. Before final loading to failure, the cylinder was partially loaded twice. The first loading was to about 1000 pounds (~16,000 psi hoop stress) to check for leaks and strain gage operation. The second loading (to about 40,000 psi hoop stress) was with the 6000 micro-strain per inch bridge sensitivity. The purpose of the second loading was to collect data for determination of modulus. The third loading was to failure with a bridge sensitivity of 30,000 micro-strain per inch of chart. The tested cylinder is shown in

Figures 45 and 46. It was evident from examination of the failure crack that a brittle failure had occurred. The failure was undoubtedly initiated in the area of the bulge near one end of the cylinder. The mechanical properties of the steel cylinder as measured by the hydraulic test are as follows:

Tensile strength: 83,100 psi

Young's modulus: 29.7×10^6 psi

Yield strength (0.2% offset): 78,000 psi

Elongation at Failure: 1.4%

Except for elongation the measured values compare closely with the stated literature values. The elongation value is believed to be low for two reasons:

(1) the failure (area of maximum elongation) was not in the area of the strain gages

(2) because of the amount of machining done on the cylinder, the steel was probably work hardened so that normal elongation was not achieved.

The stress-strain curves for the two bridge sensitivites are shown in Figures 47 and 48. The strain data for three of the gages were practically identical so these curves are represented by gage E in Figure 47 . The other curve represents strain data from gage W. The modulus of the cylinder was calculated from the stress-strain plot shown in Figure 48 which was plotted with the lower strain sensitivity readout. The hoop tensile strength and yield strength was derived from the stress-strain plot shown in Figure 47 which was plotted with the higher strain sensitivity readout.

GTC 297-2

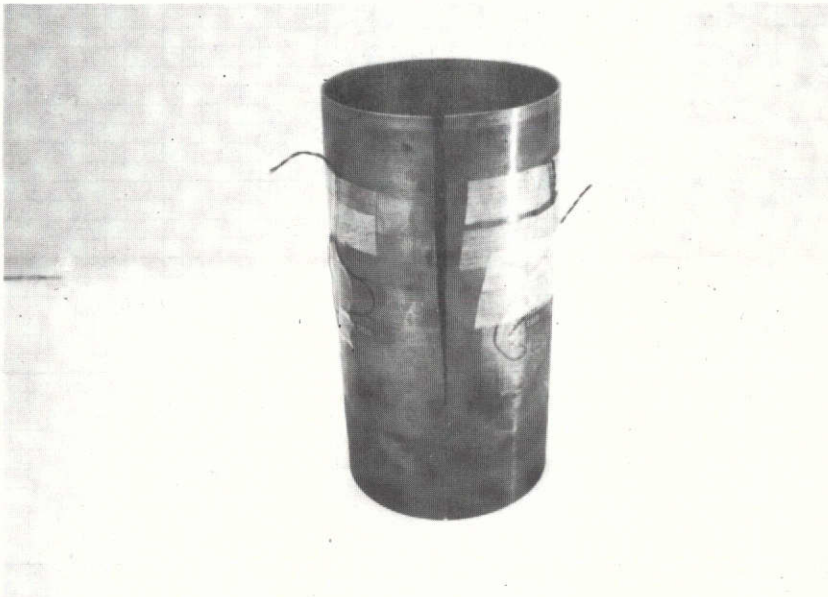

Figure 45. **Burst Tested Carbon Steel Cylinder Showing Axially Mounted Strain Gages**

GTC 297-3

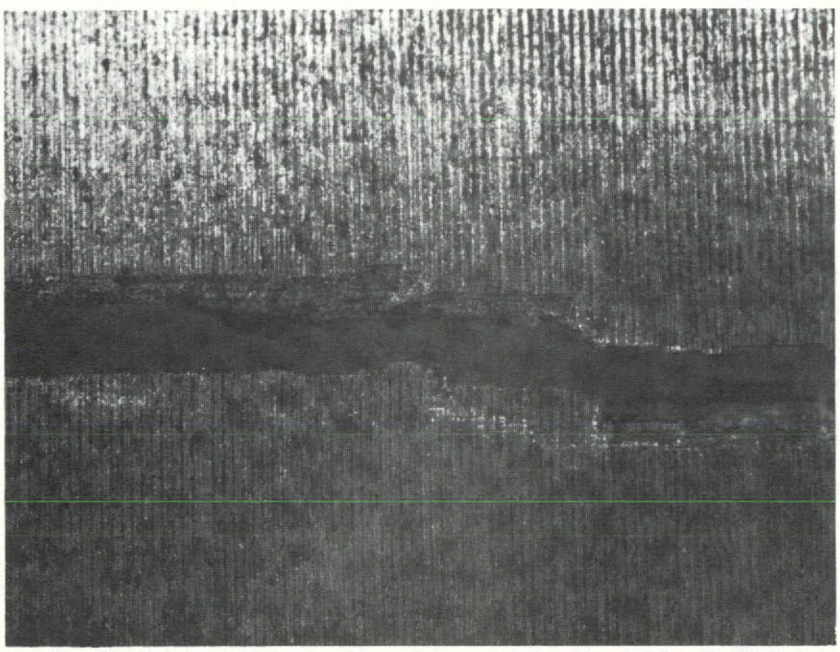

Figure 46. **Fracture Surface of Burst Tested Carbon Steel Cylinder Showing Brittle Fracture, 8X**

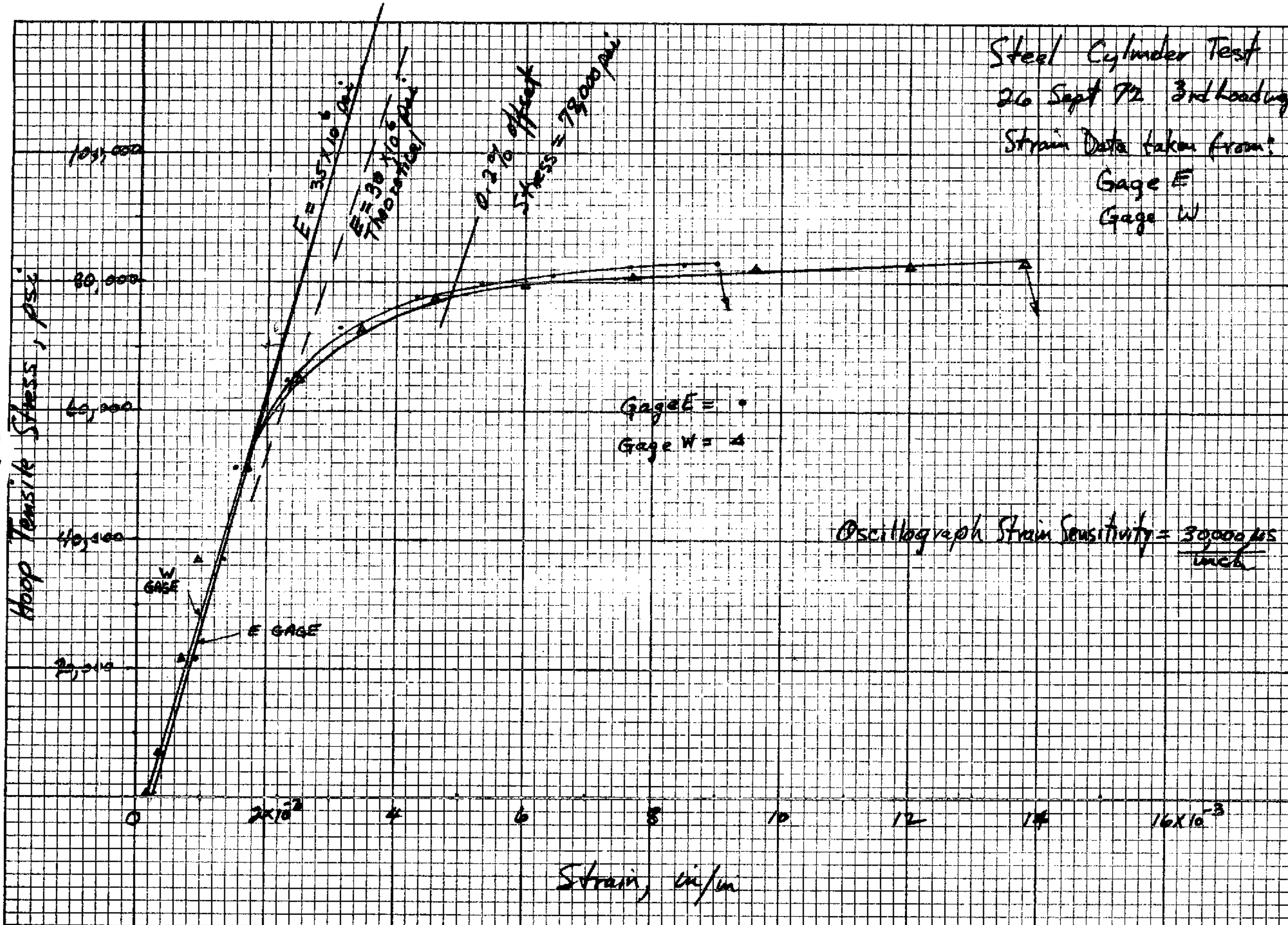

Figure 47. Stress-Strain Plot For Steel Cylinder Burst Test

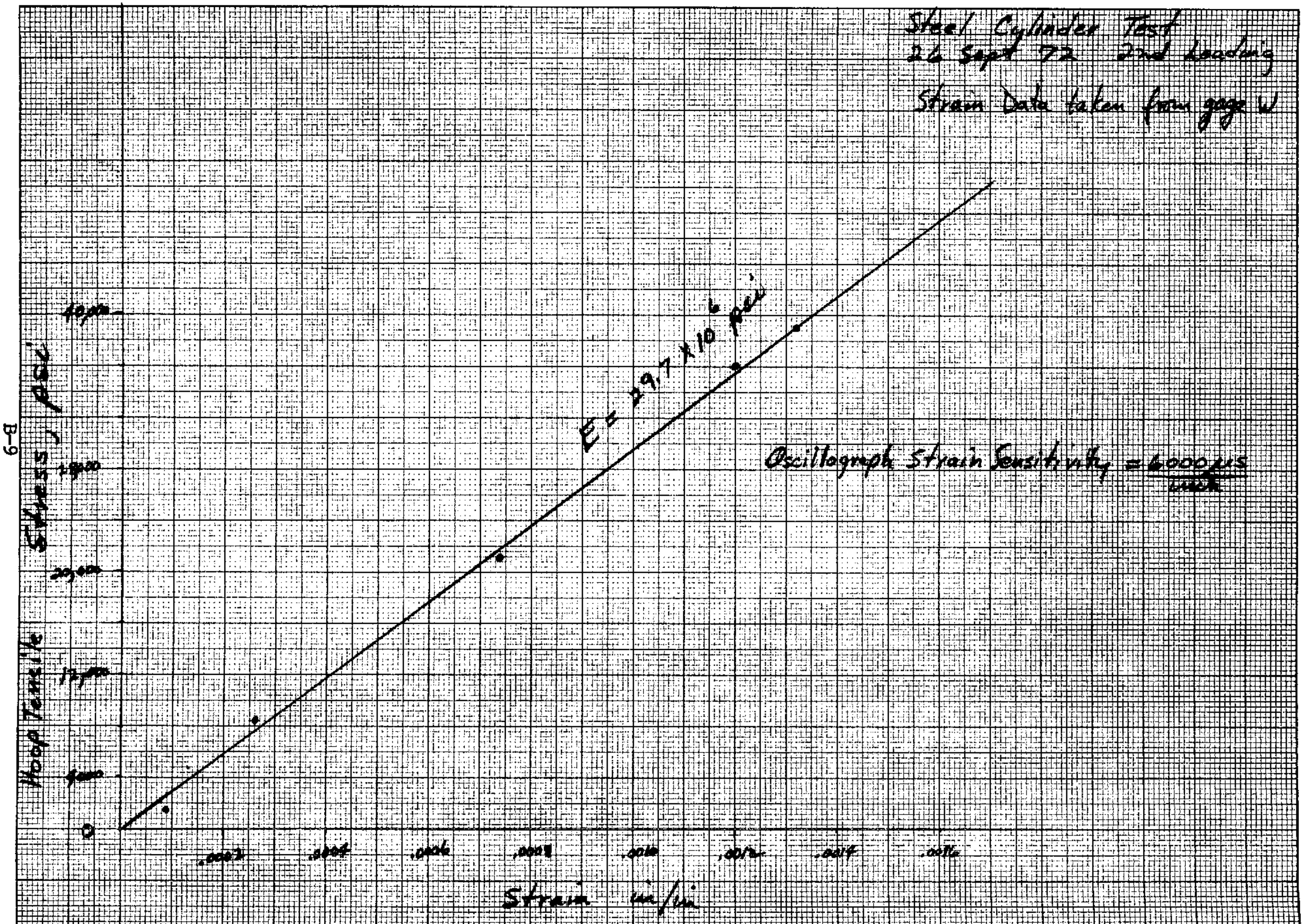

Figure 48. Stress-Strain Plot For Steel Cylinder Burst Test

B. RESULTS OF STRAIN GAGE MEASUREMENTS DURING TESTING

The strain gage read-out data was plotted in the form of stress-strain curves for those tested cylinders where sufficient data existed and are shown in this section.

Figure 49. Stress Strain Curve for Cylinder No. 7

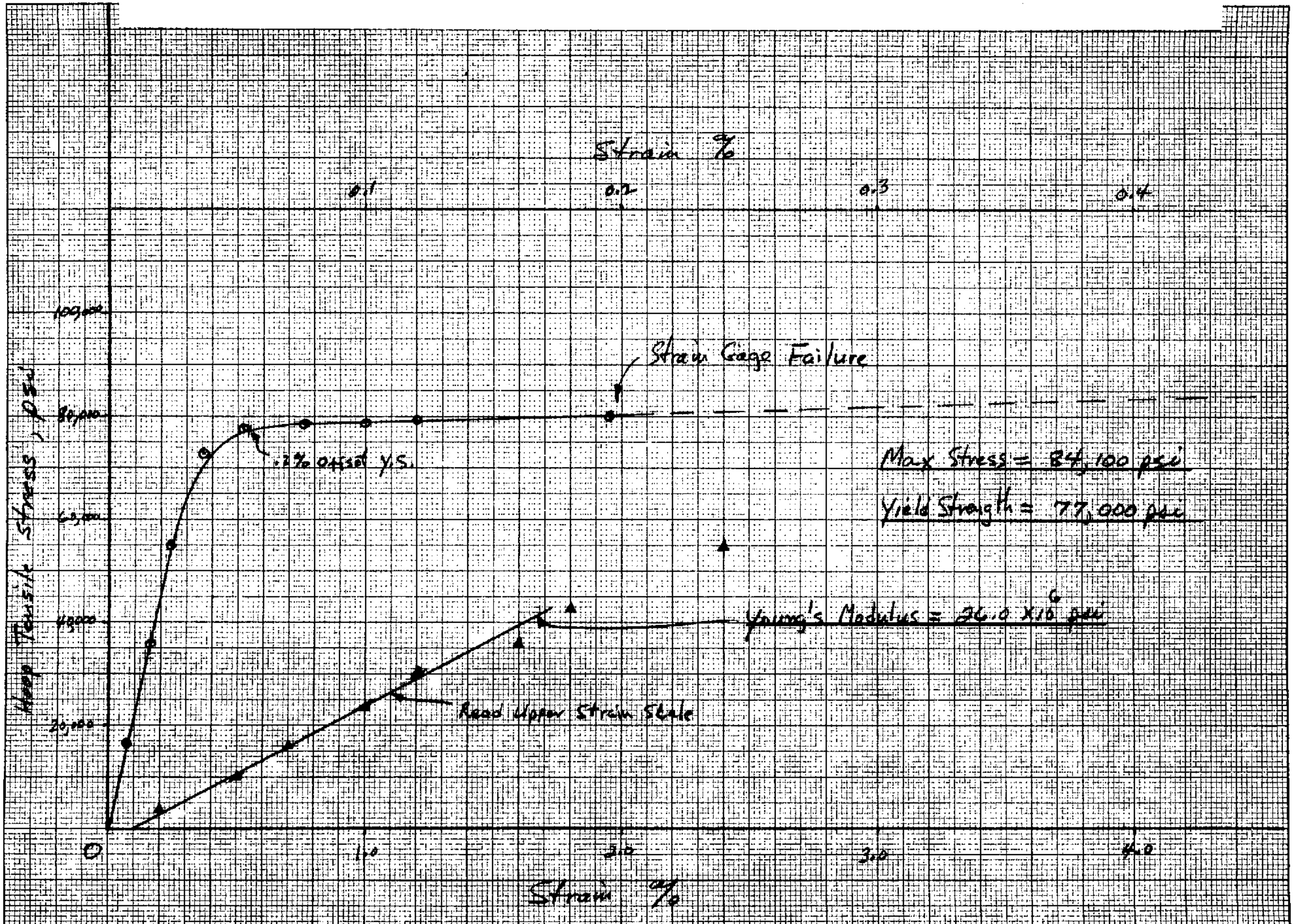

Figure 50. Stress Strain Curve for Cylinder No. 8

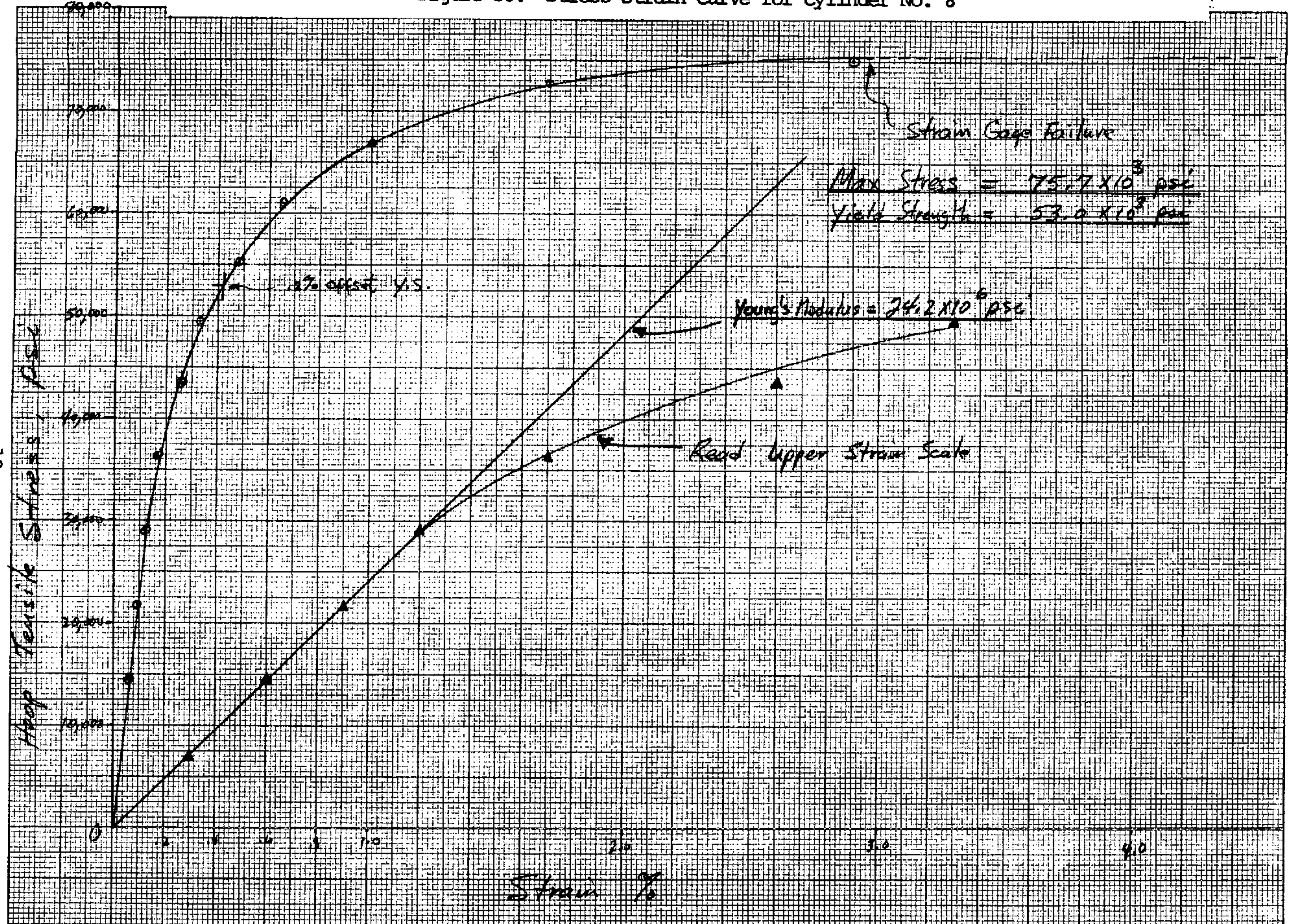

Figure 51. Stress Strain Curve for Cylinder No. 9

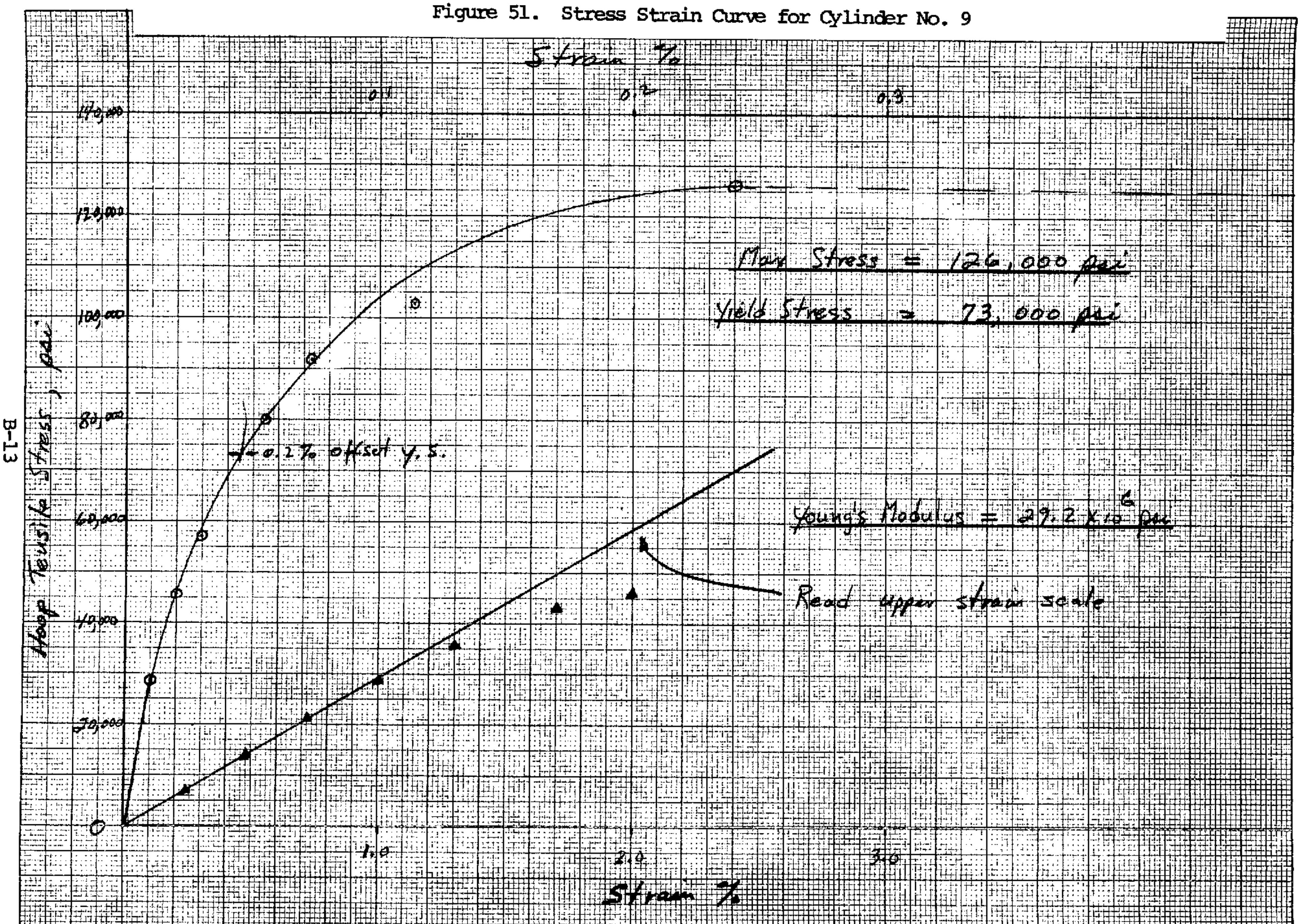

Figure 52. Stress Strain Curve for Cylinder No. 10

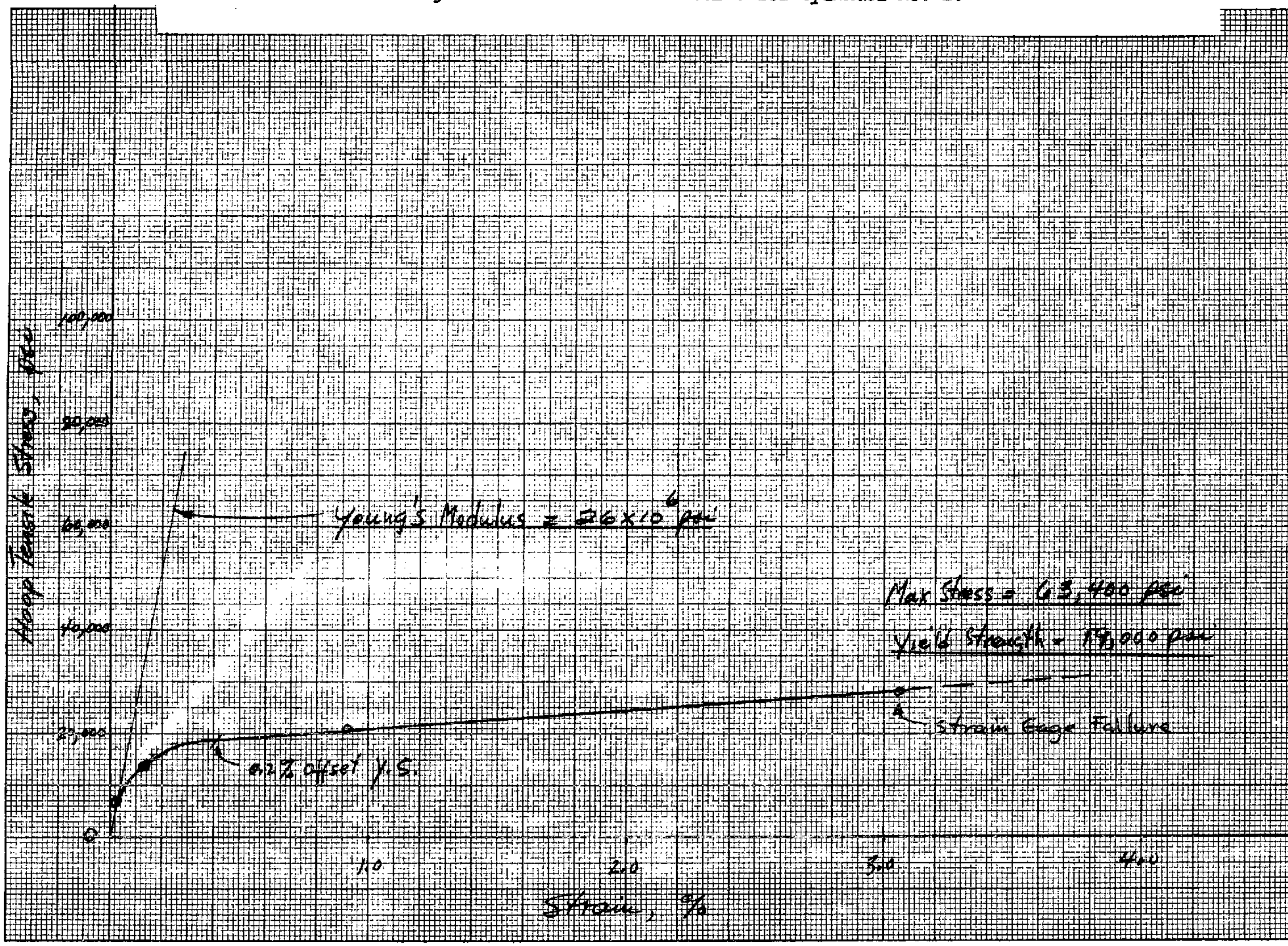

Figure 53. Stress Strain Curve for Cylinder No. 12

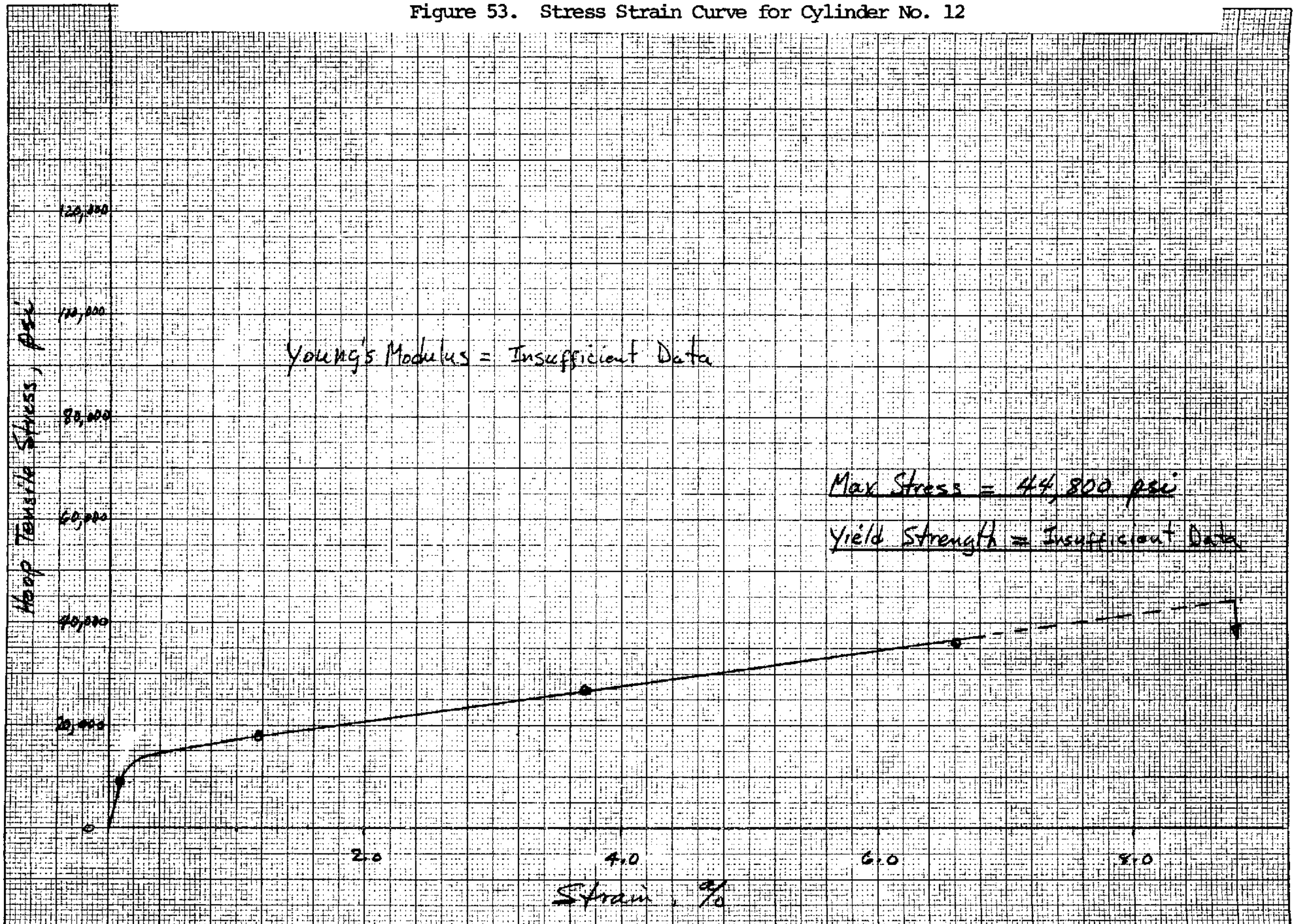

Figure 54. Stress Strain Curve for Cylinder No. 13

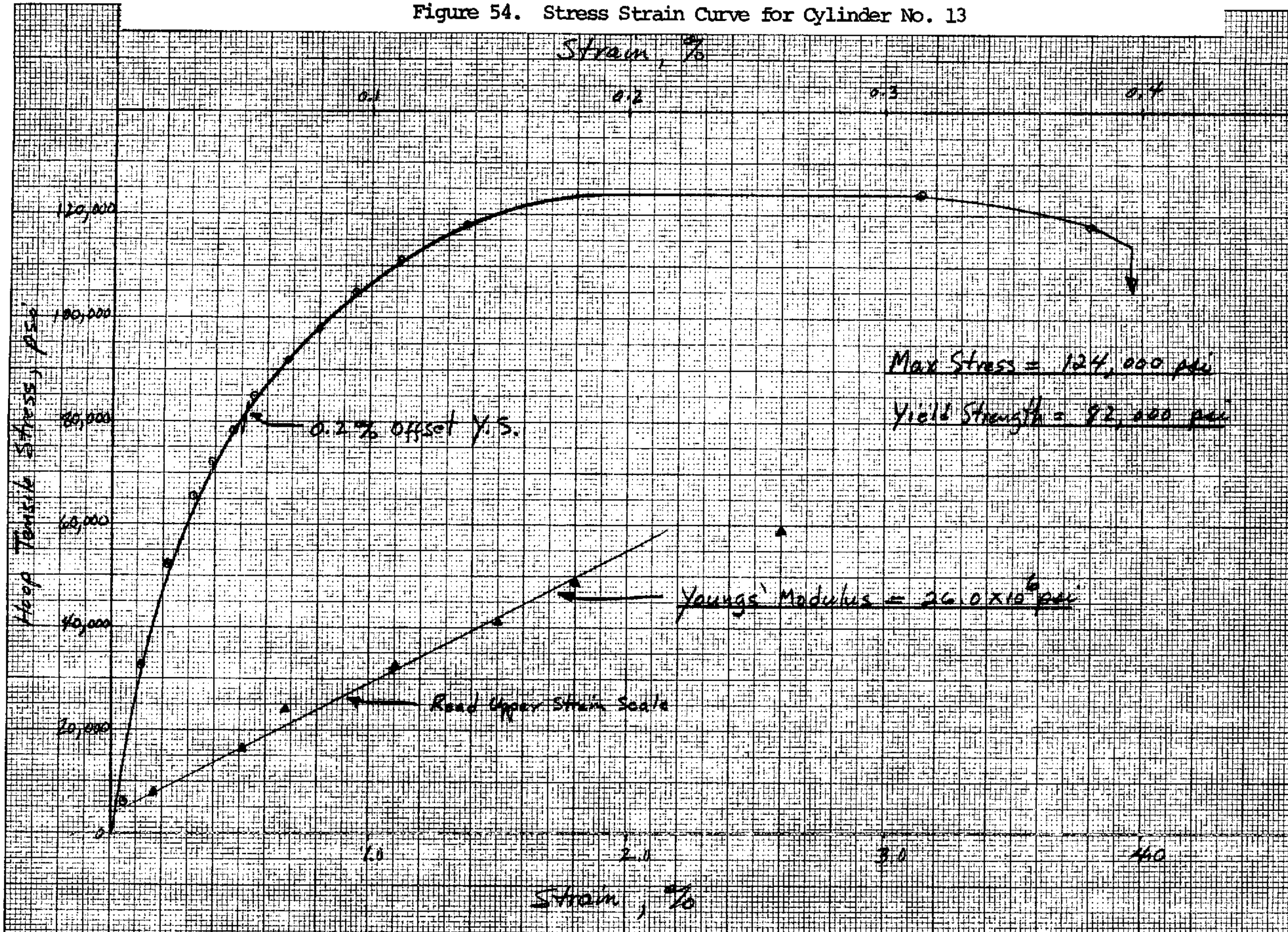

Figure 55. Stress Strain Curve for Cylinder No. 14

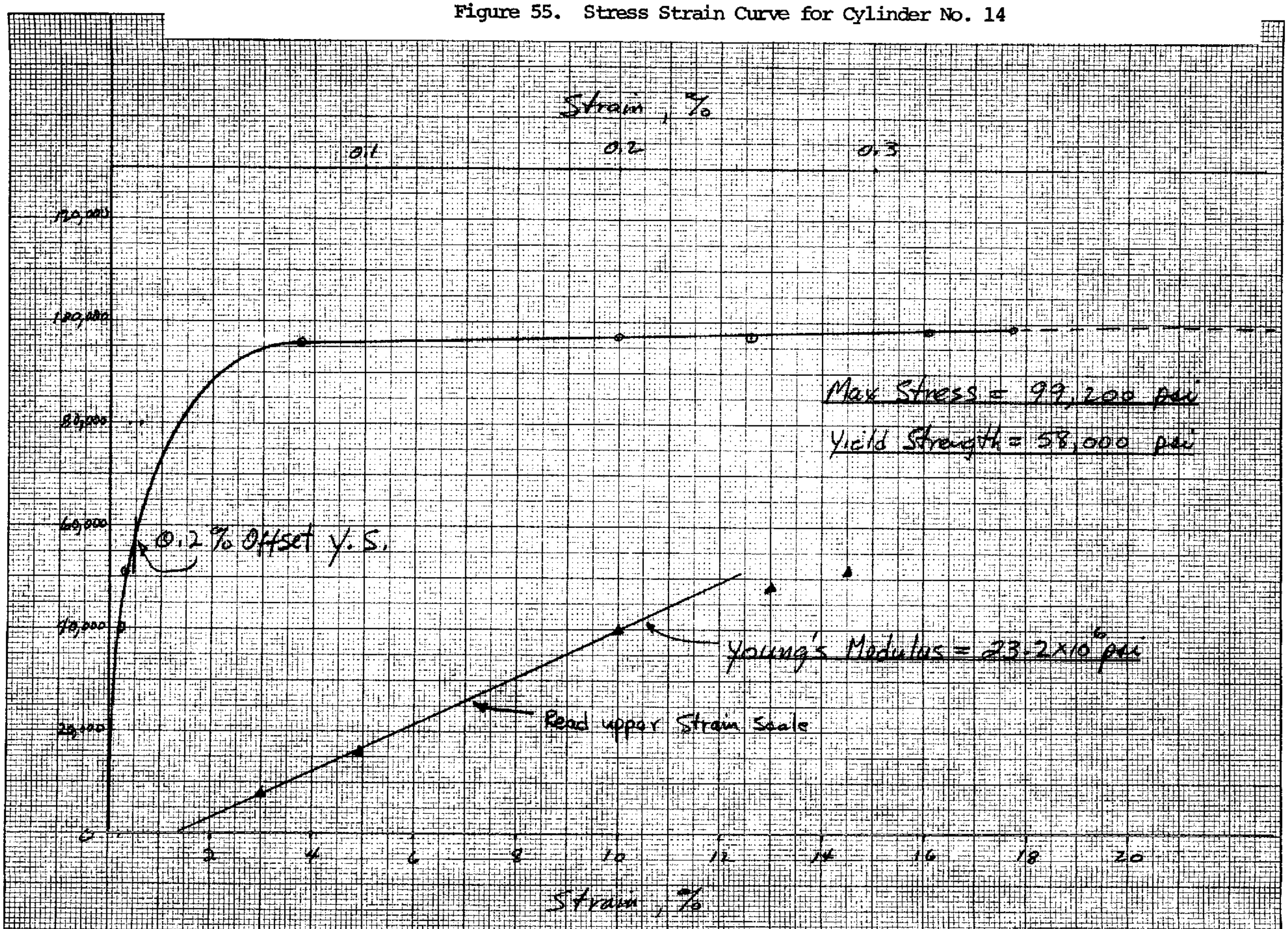

Figure 56. Stress Strain Curve for Cylinder No. 17